PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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Applicant:	David Wollan
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Title:	Alcohol Reduction in Beverages
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Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

FIRST DECLARATION OF DAVID WOLLAN UNDER 37 C.F.R. § 1.132

I, David Wollan, declare:

1. I am the inventor named in this patent application. I am a director of Memstar Pty Ltd.

4. Even our commercial competitors have recognised the unique nature of the Memstar technology. At the 2010 Intervitis Interfructa International Congress in Stuttgart, Germany, a paper by Dr Ferrarini (attached) was presented by E Bocca, a representative of Vason Group. This Italian company has commercialised an application of the contactor process as described in the Michaels patent. In the Ferrarini paper, various membrane dealcoholisation technologies are assessed. In particular the direct contact process is explained and a mass of data on flavour differences is presented. Notably, the authors recognise that this process leads to significant volatile flavour loss, and so they propose a number of alternative combined membrane configurations to overcome this. Slide 20, titled "1 To avoid loss of flavour", shows two combinations of OI (Reverse osmosis) or NF (nanofiltration) and CT (contactor). In the slide presentation to the congress on that day, the arrangement on the left was referred to as "Vason Patent" and the arrangement on the right "Memstar Patent". The slides make clear that Vason also recognises the limitations of the direct contact (Michaels) process and has accepted that Memstar's approach overcomes these.

In Ferrarini's slide "3 To improve yields" he illustrates the further stages of heating the nanofiltration permeate to 65C prior to dealcoholisation and cooling it back to 20C prior to recombination, as in my claims 7, 8, and 36-38. This is a specific recognition of the improved performance of the process with heating in these claims.

5. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true. I understand that willful false statements and the like if made herein would be punishable by fine or imprisonment, or

both, under section 1001 of Title 18 of the United States Code, and may jeopardize the validity of the application or any patent issuing there from.

David Wollan

Date: 24 January 2011

Attachments:

"Memstar Wows Judges with Competition Triumph," *Australian & New Zealand Grapegrower & Winemaker*, July 2007, pages 60-61.

2010 Intervitis Interfructa International Congress in Stuttgart, Germany, paper by Dr Ferrarini (presented by E Bocca)

Memstar wows judges with competition triumph

John Hudswell

As proud sponsors of the inaugural Wine Industry Suppliers Association (WISA) WOW Award, *Grapegrower & Winemaker* congratulates Memstar as the winner with the company's Alcohol Adjustment (AA) innovation. The prize? A trip to the SIMEI exhibition in Milan in November 2007.

The ability to lower alcohol levels in wine is important for the wine industry and Memstar was able to demonstrate that in the past six months with about 50 million litres of wine treated through the AA process. In 2005-06 Memstar processed 5-6 million litres so in terms of a measurable and demonstrable WOW factor, and impact on the wine industry, that growth is what got Memstar the judge's vote.

The quest to please the palates of winelovers is never ending. Some like robust reds, while others prefer a fruity white, and there are some who prefer wines that don't pack such an alcoholic punch.

That was the cue for Memstar to become acutely involved in the wine industry.

It embarked on the trail of lowering the alcoholic level of wines but without lowering the quality and it found the answer with the innovative use of a specialised membrane.

In the past two years the technology has rapidly been embraced by winemakers around the world. In the past year the technology has been used in Australia, New Zealand, United States (Oregon, California), Chile, and South Africa. In June the technology was undergoing trials in France and similar trials are scheduled to be held in Argentina.

"The technology enables the energy-efficient reduction of alcohol in wines without heating the wine, Memstar business development manager Tim Grosser said "It is done entirely onsite with minimal wine volume reduction. "It reduces alcohol concentration without damaging flavour and aroma compounds. "It is an economic process and the energy consumption is very low compared to traditional alternatives."

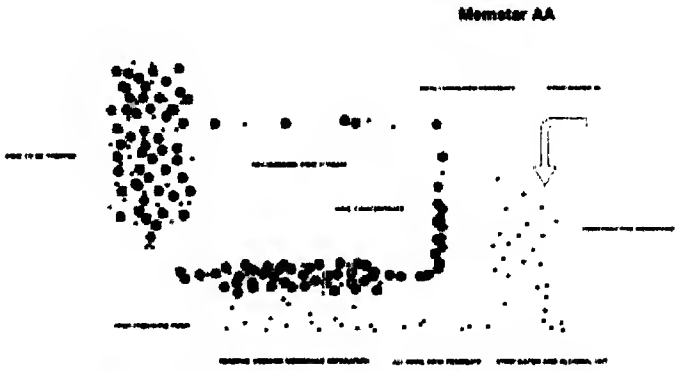

Fig. 1. Schematic diagram of the Memstar Alcohol Adjustment process.

The technology makes innovative use of a specialised membrane.

"This membrane is a hydrophobic hollow fibre or perstractive membrane," Grosser said.

"Memstar applied this technology to use a strip water to remove alcohol from wine RO permeate (liquid to liquid). "By applying RO permeate to this perstractive membrane the membrane performs very well with very infrequent cleaning required and stable performance.

"This approach also ensures that, essentially, only alcohol is removed from the wine with no loss of flavour or aroma compounds," Grosser said. Memstar optimised the technology by removing oxygen from strip water to prevent oxidation of wine and warming of wine permeate to enhance the capacity and economics of the process.

Over the past 20 years there has been a steady increase in the mean alcohol level of wines from 12.4% in 1984 to 14.2% 2002 (analysis by the Australian Wine Research Institute).

This trend stems from winemakers' attempts to avoid unpleasant green fruit characters by leaving their grapes to hang longer as they strive for more mature flavours and tannins and softer acidity. The resultant higher sugar and hence high alcohol levels have been an unwelcome but necessary consequence of this pursuit of greater ripeness.

"Until the development of Memstar AA, winemakers had few acceptable options for dealing with this dilemma," Grosser said.

"The patented Memstar AA process is a 'closed loop' with two membrane barriers between the wine being treated and the strip water which removes the alcohol. "The actual alcohol stripping step is performed on reverse osmosis permeate which contains very little of the precious wine characters that would otherwise be lost. "The process is carried out in a closed, oxygen free environment and does not involve the addition of any extraneous material."

He said the process did not involve the addition of water to wine – a practice that is not permitted in most

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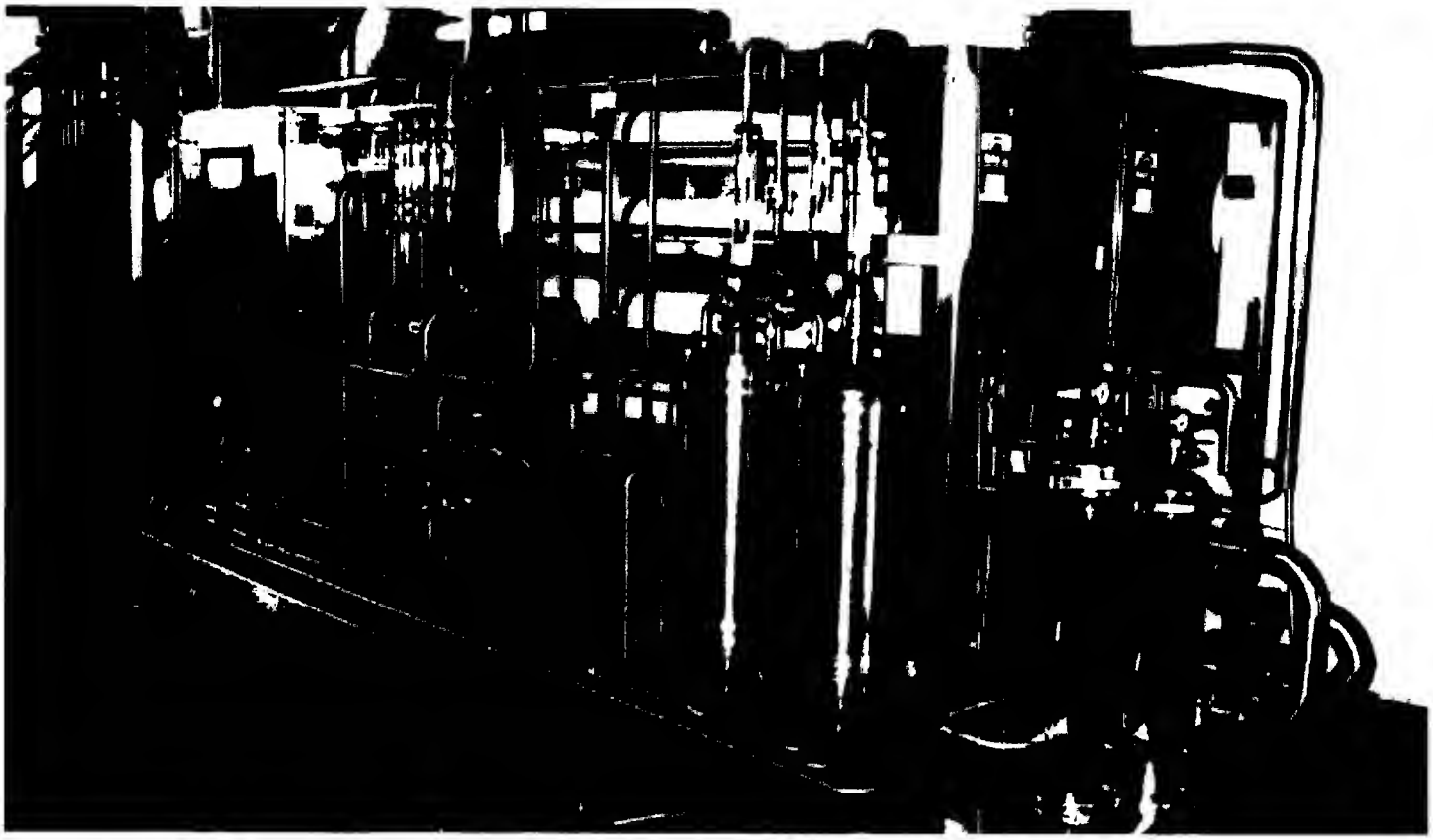

Fig. 2. A Memstar Alcohol Adjustment machine capable of over 200 litres of alcohol (LAL) removal per hour. Note the portability of the one skid containing all of the equipment.

wine-producing countries. It also avoids the legally dubious addition of water derived from other grape sources.

Grosser says the Memstar system allows the winemaker to perform 'sweet spot' trials at the commencement of the process and as it proceeds. "By monitoring progress as the 'sweet spot' is approached, winemakers can stop the process at their preferred alcohol level," Grosser said. Alcohol has a profound effect on a taster's perception of a given wine's quality and style. When a wine is reduced in alcohol without changing any other components, there are certain alcohol concentrations where the wine seems to stand out, displaying enhanced fruit expression on the nose and palate and improved mouth-feel. These points are called sweet spots. What is not so well appreciated is that varying the alcohol level by as little as 0.1% may be the difference between a sweet spot or not. Also, varying the alcohol can have a profound effect on the perceived style of a given wine. Depending on the alcohol content, for a given wine there may be more than one appealing sweet spot, corresponding to quite different styles.

A trend to slightly lower alcohol wines in the past four or five years prompted Boar's Rock to take a closer look at the Memstar system. A Memstar unit was purchased and installed at its McLaren Vale site early this year, and, according to managing director, Mike Farmilo, initial trails have been successful!

He said many customers wanted to bottle at 14-15% alcohol, but harvesting at high baume, and with good sugar/alcohol conversions, their wines were generally ending up over 15%. The Memstar unit allows customers to reduce the alcohol to what tastes best for them, and to achieve a better balance of flavour and alcohol. Apart from the fact that the taste trend is changing, those who export wines have to be cognizant of taxation laws that come into effect when the alcoholic level is above 14%. Farmilo said the Boar's Rock unit is portable so that it can be used at its other two sites, Waikerie and Langhorne Creek. He said operation of the new system was "reasonably simple' and Memstar had provided a good training and backup service.

The Memstar AA alcohol adjustment process is available as a cost-effective, on-site service and is available on a short or long term hire basis or may be purchased outright.

Memstar will be at WineTech in Adelaide beginning 29 July, so pay them a visit.

Memstar provides Boar's Rock with greater flexibility

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The WOW Award proudly presented by WISA Wine Industry Suppliers Australia Inc. Australian & New Zealand Grapegrower & Winemaker

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CONGRESS-PROGRAMME 2010

INTERVITIS INTERFRUCTA

Quality · Sustainability · Marketing: Impact on Innovation

5:25 p.m. **A Microbiological Systems Comparison between Integrated, Organic and Biodynamic Plant Treatment Strategies**
Dr. Christian von Wallbrunn, Forschungsanstalt Geisenheim, Germany

5:50 p.m. **Organic Wine Production: Implementing Oenological Rules on EU-Level**
Dr. Uwe Hofmann, ECO-CONSULT, Germany

6:00 p.m. **End**

PROCESS TECHNOLOGY FOR PROCESS CONTROL AND ALCOHOL MANAGEMENT

Moderation: Prof. Dr. Monika Christmann and Dr. Gerd Scholten

2:00 p.m. **Poster Session**

3:00 p.m. **Introduction**

3:05 p.m. **Direct Grape Processing and Juicing in the Vineyard**
Prof. Dr. Tilo Hühn, ZHAW Zentrum für Getränke und Aromaforschung, Switzerland

3:25 p.m. **Carboxymethylcellose for Crystal Stabilisation**
Dr. Gerd Scholten, DLR Mosel, Germany

3:45 p.m. **On-line Measurement of Electrochemical Potential on Must and Wine: Applications and Examples in Wineries**
Mariano Fernandez, DOLMAR, Spain

4:05 p.m. **Coffee Break**

4:20 p.m. **Sensory Impact of Ethanol – the Hidden Sensory Giant**
Prof. Dr. Ulrich Fischer, DLR Rheinpfalz, Germany

4:40 p.m. **New Membrane Approach to Optimise the Concentration of Dissolved Gases in Wine and to Reduce the Alcohol Levels of Wine**
Dr. Oliver Schmidt, LVWO Weinsberg, Germany

5:00 p.m. **Technological Options for Dealing with High Alcohol Wines**
David Wollan, Memstar Pty Ltd, Australia

5:20 p.m. **Many years' Experiences on the Dealcoholization Processes Using Membranes Techniques to Guarantee the Quality and Sustainability of the Wine**
Prof. Roberto Ferrarini, Università degli Studi di Verona – Dipartimento di Scienze, Tecnologie e Mercati, Italy

5:40 p.m. **Tasting**

6:00 p.m. **End**

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Internationale Technologiemesse für Wein, Obst, Fruchtsaft und Spirituosen
INTERVITIS INTERFRUCTA
Neue Messe Stuttgart
24.–28. März 2010

Many years' experiences on the dealcoholization processes using membranes techniques to guarantee the quality and sustainability of the wine.

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Abstract

In this work are shown the results of several experiments performed with different membrane techniques (contactor, osmotic membranes, nanofiltration) on different white and red Italian wines with different degrees of alcohol removal, reporting chemical analysis of the major and more quality and stability related components of wine (varietal and fermentative aromatic fractions), as well as performances data of the various tested membrane processes.

Introduction

Currently it is increasingly common to find an excessive alcohol content in wine production, due, first, to the climate trends of the last harvests that led to this effect even in the temperate regions. Also the search for advanced states of ripening of grapes, aimed at obtaining structured wines with a strong territorial characterization, can result in an excessive presence of sugars in the musts and, consequently, of alcohol in the wine; such situation frequently affects high-range wines.
The excess of alcohol content, which more and more frequently can exceed the limit of 15%, does not guarantee the qualitative equilibrium, and represents for the consumer the most critical factor in assessing the product's wholesomeness.
To overcome this critical issue, the European Union with the entry into force of the new CMO, has allowed wines' alcohol removal through physical processes for up to 2 alcohol percent. On the contrary in the United States the problem of alcohol excess is resolved by adding water directly to the musts with high sugar content (over 22 ° Brix), thus restoring that water due to dehydration of the berry before harvest. This technique has different qualitative effects compared to the alcohol removal performed on the wine, and it may also be less culturally acceptable by the consumers and the producers of the more traditional wine areas such as the European ones.
Concerning alcohol removal techniques, there are different technological approaches: decreasing must's potential alcohol content (removal of sugar concentration), or separating a fraction of the alcohol from the wine.

In this case the reduction of wines' alcoholic content can be made with different techniques of physical separation: Spinning Cone Column, distilling the RO (reverse osmosis) permeate, pervaporation, use of "Contactor" membranes.
In particular, the "Contactor" membrane technique, by itself or combined with other separating actions, enables the implementation of highly performing alcohol removal processes, some of which have been researched and tested (the relative results are reported below).
Moreover, the aim of this investigation is the control of the volatiles content variation due to these technological test and possibly justify the maintaining of several quality-supporting compounds that, according to the actual literature knowledges, can be important and characteristic of some famous Italian wine types, like a fresh fermented white 'Moscato d'Asti' – both as still and sparkling wine in different experimental approaches -, two three-year old still red 'Sfursat' wines from partially dried Nebbiolo grapes of Valtellina area in Northern Italy close to the Switzerland, a two-year old Chianti of Tuscany and a two-year old Valpolicella of Verona Province.

Materiali e metodi

Dealcoholization system

The dealcoholization technique studied in this work is the "Contactor"; it involves the treatment of wine with hydrophobic membranes that, used between the product and an extractive solution (water), creates a gaseous layer (gaseous membrane) trough which the most volatile compounds pass. Among these compounds, the most representative is alcohol (Fig. 1). The difference between the vapour pressure of alcohol in wine and the extractive solution permit the passage of ethanol, in form of vapour, trough the gaseous membrane and then the dissolution in the water of the extractive circuit.
The membranes used in this work have been supplied by Juclas (Verona – I); they're PTFE (Teflon®) hollow fibers (ext. Ø 1 mm, thickness 200 µm).
The system used for the study of the effects of dealcoholization on wine composition is a pilot plant with a 0,2 m^2 membrane; the industrial plant, used to remove up to 2% of alcohol, has a membrane with the surface of 20 m^2; the flow rate of wine and water in contact with the membrane is of 0,1-0,3 ms^{-1}. All the treatments have been carried out at environmental temperature. The vaporizing efficiency of the membrane is between 0,1 and 0,2 l $m^{-2} h^{-1}$ of anhydrous acohol at 20°C .

Study of the effects of the dealcoholization rate with a contactor membrane on the composition of wines.

The experiences have been made just after the end of fermentation on wine from Moscato Fior d'Arancio grapes, produced in the zone of Colli Euganei, Padua (I).
We performed four dealcoholization experiences at 4 different alcohol removal rates: 1.4 , 2.7 , 5,1 and 7,1 % v/v.
Industrial tests of low dealcoholization (up to 2% removal) on some typical Italian wines.
Considering the recent legal changes with the new OCM, it's allowed a dealcoholization up to 2% v/v.
Here are reported some dealcoholization experiences lowering the acohol degree up to 2° (maximum allowed by the European law) of some typical Italian wines: a red wine

"Valpolicella" (produced in the Verona region), 2 "Sfurzat" from Valtellina (Sondrio region), 1 "Chianti" produced in Tuscany (Siena).

Analytical methods

Analytical determination performed on wines have been made following the European [CEE, 1990] and OIV [OIV,2007] methods.
It has been evaluated tartaric stability by the T°sat and the Mini contact methods using the Criosmall instrument (Steroglass – Pe – l).
Aroma profiles have been investigated after proper enrichment techniques due to the substances and/or relevant concentrations we are going to consider, like SPE (Solid Phase Extraction) using a very exhaustive adsorption cartridge (ENV+), but mostly with headspace-SPME (Solid Phase Microextraction) with a three-phases fiber [see some literature references and some recently optimised methods by UIV Co., Verona]. All the compounds enriched have been quantified by HRGC-MS. In few cases we considered also the quantification of methanol, acetaldehyde and ethyl acetate by direct injection in GC-FID, after a preliminary distillation, 1:4, v/v, of the matrix (capillary column CPWAX-57, 30 m x 0.3 mm x 0.32 µm). Acetaldehyde and ethyl acetate could contribute in fact to some off-flavour as a grassy and vinegar-like note, respectively.
Analytical attention has been payd mainly to the following compounds classes:
- secondary compounds – the very important quality-imprinting contributors -, like fermentation esters (acetates of higher alcohols and other alcohols; different ethyl esters, mostly the fatty acid ones from C4 to C10), the so-called 'leaves' C6-alcohols, some peculiar fermentation scent-active compounds like 2-phenylethanol and methionol, some derivatives from hydroxycinnamic acids as vinyl- and ethylphenols;
- varietal compounds, basically: monoterpenols, monoterpenes, monoterpenyl oxides, monoterpenyl ethylethers (usually in traces, but potent aroma contributors!), norisoprenoids (C13-carotene derived compounds) and some aryl compounds;
- different storage/ageing compounds mainly derived from the barrels and relevant co-operage techniques.

Sensory Analyses

Moscato wines, grown from study of the effects of the dealcoholisation, have been sensory analyses using 27 trained panelists. The descriptors chosen for sensory analysis were: bitter, sweet, salty, acidity, fruity, floral, honey, vegetable, structure, persistence. Data were collected through a "unipolar non-structured" scale (M. Ubigli, 2004) with values from 0 to 100. Results were treated statistically with Friedman test.

Results and discussion

Study of the effects of the deacoholization rate with contactor membrane on the wine composition

The main composition of the wines analyzed for the study of dealcoholization effects are reported in Tab. 1.

Data show, as predictable, an increasing of the fix components of wine which is proportional to the concentration due to the ethanol removal and consequent volume reduction.
In Table 1 are also reported the Tsat test results of the dealcoholized wines; the Tsat decreases proportionally to the dealcoholization rate.
The following sentences regards the aroma compounds: (Tab 2)
A quite linear decrease happens both for the acetates (ethyl acetate enclosed even if with a partially quantittive evaluation) and the ethyl esters of fatty acids reaching about 1/5 content for C4-C8 acid esters and about 1/10 for C10-ester. This can cause an important variation in the fruity scent of the wines. On the opposite, the relevant acids do not significantly variate.
Also the C6-alcohols seems to decrease proportionally with the proof lowering.
As important varietal compounds, mostly responsible of the floral scent above all in the case of Muscat wines, also a very little decrease roughly of 5-10% both in single compounds and in the sum could be estimated. This also for the 4-terpineol, monoterpenol connected to the intensity of the drying phase.
For the monoterpenols, we have a quite linear important decrease above all for linalool with an initial decrease and a subsequent increase for citronellol, nerol, and geraniol while α-terpineol tends always to increase. These different situation should be judged by considering that α-terpineol is also a chemical transformation compounds from linalool, nerol and geraniol, and that other variations can be justified with a longer time of the treatment, therefore favouring chemical hydrolysis of the bound forms and possible secondary metabolic reaction as that of citronellol yeast formation from nerol and geraniol.
Excluding the 2,7% dealcoholization data, the β-damascenone trend is slightly increasing due to the concentration effect of the technique and to the possible improved hydrolysis of the precursors.
The vinylphenols and benzaldehyde seems to have a strange evolution situation.

Industrial low dealcoholization (up to 2%) experiences on tipical Italian wines

We compared the variation among the wine that comes from industrial experience of limited Reduced Alcohol wine, with different alcohol removal degrees, as written in columns heading (Tab 3).
We observed a decrease from ethyl esters (iC5, C4-C10) similar among the wines varying between 60 and 87%, tendencially a few higher for the MW-higher esters. An exception is represented for the MW-lower esters in the case of Chianti, likely due to a limited fermentation by Brettanomyces in the dealcoholized sample favoured by the reduction of antiseptic effect of alcohol and by the possible airing occurred during the treatment.
Also the relevant acids presented a limited decrease in the case of Sfursat wines and Chianti (meanly at about 20%), but a rather constance or a few increase in the other cases.
As for the acetates – also important in contributing to the fruity scent -, both the most representing one, the isoamyl acetate, and the total sum of them have a level reduction of about 20%.

For the alcohols 2-phenylethanol, methionol and benzyl alcohol we can observe a small decrease from 5 to 20%, a little higher for methionol except for the Valpolicella wine with a possible alteration due to the Brett metabolism.
As aroma interesting trace-compounds grouped as 'phenols' like eugenol, guaiacol, o- and p-cresol and phenol, when properly evaluated as a sum in our wines, even a possible decrease of about 20% can be estimated. This also for the oak lactones, however with a quite constance in the case of Valpolicella wine.
In the case of C6-alcohols, a little decrease can also be observed at about 10%.
As important varietal compounds, a very little decrease roughly of 5-10% both in single compounds and in the sum could be estimated. This also for the 4-terpineol, monoterpenol connected to the intensity of the drying phase.
Finally we considered some norisoprenoids which are possible complementing scent in the wine just as young product, as the β-damascenone and β-ionone, or by progressed aging as TDN (1,1,3-trimethyl-1,2-dihydronaphthalene), vitispiranes (VTP), actinidols and relevant ethyl ethers. The relevant concentrations of the ethyl ethers are quite constant, while those of the compounds can have either an important decrease as for TDN until about 50% or a little more reduced variation as for VTP.
By concluding these comparison, the de-alcoholisation process limited to about 1.5 % proof could reduced the most important aroma influencing known volatiles in a similar way without a clear effect of the initial alcohol proof (please, consider the different level in the wine types considered). This reduction can variate between 5 and 30-40% with a mean of about 20%, seeming the range not depending of the wine types as from the results of the two similar Sfursat wines treated.

Sensory Analyses

The application of Friedman test revealed significant differences between divers dealcoholized rate wines for these descriptors: structure, persistence and sweet. We could note in fact a marked effect of alcohol on sensory perception of the structure and persistence, despite the increase of the extract and we can reconfirm also the important contribution of ethanol on the perception of sweet (Fig. 2).
We highlight relatively large differences, but not statistically significant, for descriptors acidity, salty and honey. However, the results of sensory analysis are sometimes controversial because they do not follow a progressive trend depending on the level of dealcoholization of the wines, so further investigations are required.

Conclusions

The testing performed and the data of industrial processes with membrane contactor can lead to the following conclusions:

- The alcohol membrane separation technique leads to a depletion of aroma compounds in the wines, especially for the most volatile like ethyl esters and lesser the acetates; these are the fermentative aromas.
- Concerning about the varietal aroma compounds it was observed a little decreasing trend on terpenic compounds; in the Norisoprenoid class the TDN presents a significant decreasing, while the β-damascenone data remains substantially unchanged.

- The de-alcoholisation process limited to about 1.5 % proof could reduced the most important aroma influencing known volatiles in a similar way without a clear effect of the initial alcohol proof (please, consider the different level in the considered wine types). This reduction can variate between 5 and 30-40% with a mean of about 20%, seeming the range not depending of the wine types as from the results of the two similar Sfursat wines treated.
- Therefore, any treatment with membrane contactor is recommended in young wines characterized by an high ester content; in case of varietal aromas wines the approach can be, knowing these results, varied depending on the volatile compounds that will characterize these wines.
- However, it is advisable to operate the dealcoholization treatment on wine fractions containing no aromatic substances.
- Finally, it should be technically noted that this technique helps to improve the tartaric stability; in contrast slight oxygenation and alcohol removal can promote the *Brettanomyces* development.
- In the dealcoholization of Moscato wines have been shown a substantial modification of the sensory profile: these wines are less structured, persistant and sweet depending on the different rates of dealcoholization; this aspect is very important to be considered in the production of wines which are well valued for the consumers.

Acknowledgements

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- Prof. Carlo Gostoli, Chemical Engineering Department, University of Bologna, Italy.
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Figures

Fig. 1 - Functioning principle of "contactor"

Fig. 2 - Sensory profiles of dealcoholized wines (* *p* of significance < 0.05)

Tables

Tab. 1 - Main chemical characteristic of wines dealcoholized at different rates by a contactor membrane.

Tab. 2 - Aroma compounds of wines dealcoholized at different rates by a contactor membrane.

Tab. 3 - Aroma compounds of industrial dealcoholized (up to 2%) tipical Italian wines.

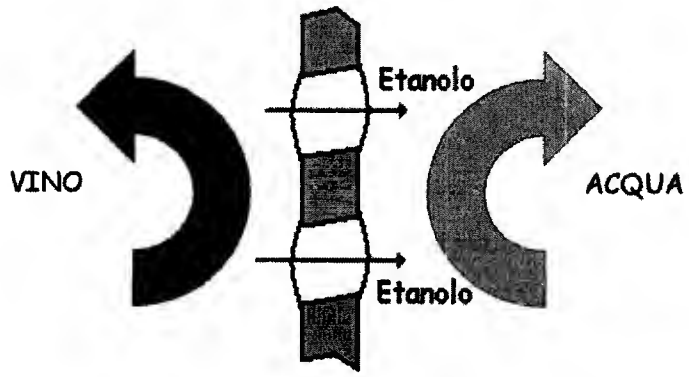


Fig. 1

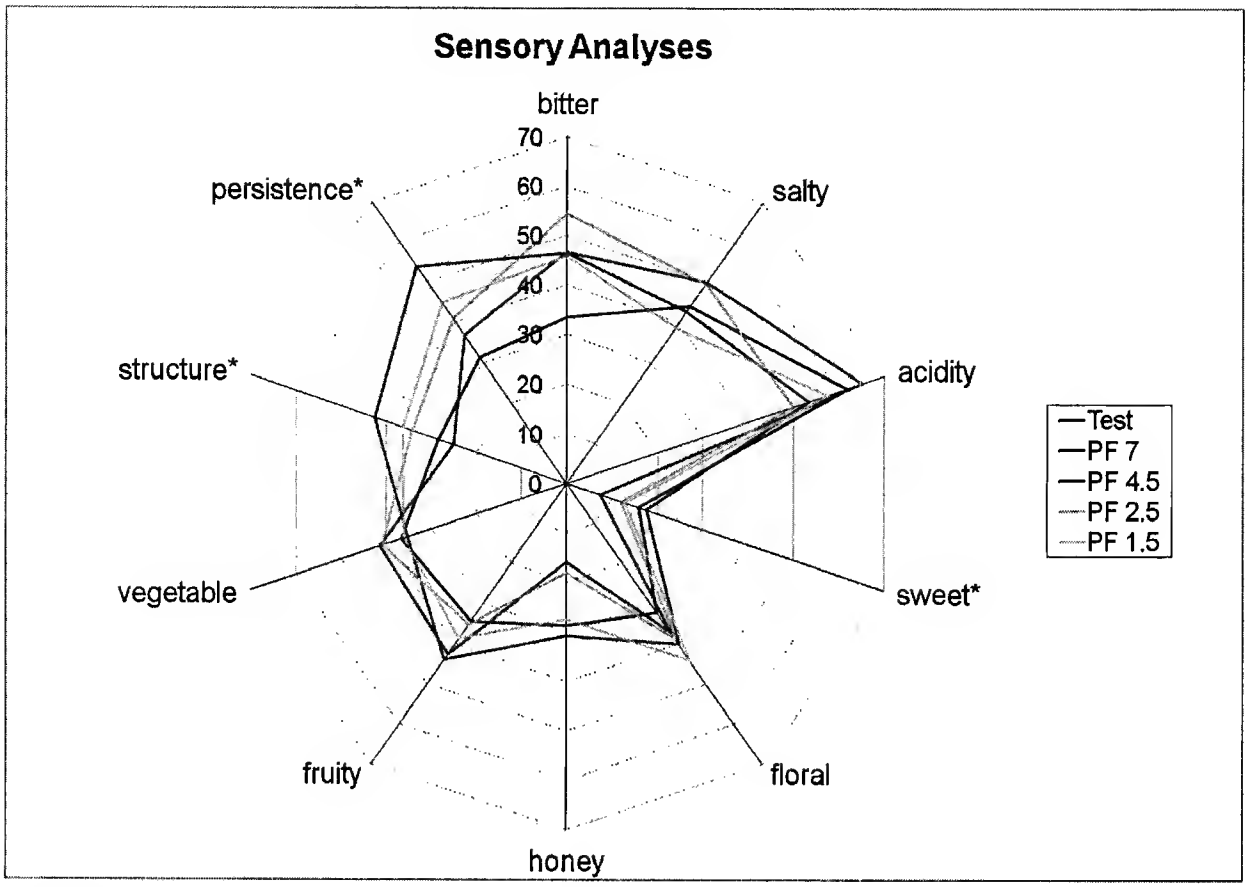


Fig. 2

% Alcohol removed		0 Test	1,4	2,7	5,1	7,1
Alcohol	**(% vol)**	12,4	11,0	9,7	7,3	5,3
pH		3,46	3,44	3,49	3,48	3,47
Titratable Acidity	**(g/L)**	6,6	6,6	6,7	6,7	6,9
Volatile Acidity	**(g/L)**	0,07	0,06	0,06	0,05	0,05
Acetic Acid	**(g/L)**	0,01	0,01	0,01	0,01	0,01
Total exctract	**(g/L)**	23,5	23,9	23,2	24,2	24,7
Glycerol	**(g/L)**	10,1	10,8	10,8	11,1	11,5
Tartaric acid	**(g/L)**	1,82	1,79	1,90	1,88	1,89
K	**(mg/L)**	638	664	653	675	709
Ash	**(g/L)**	1,89	1,80	1,81	1,81	2,17
Ash alkalinity	**(meq/L)**	20,23	18,8	19,0	21,0	21,3
Saturation temperature	**(°C)**	22,1	20,4	20,1	17,8	16,5

Tab. 1

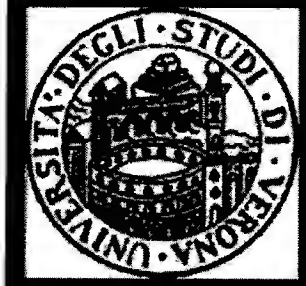
COMPOUND CLASS	COMPOUNDS TESTED FOR SPME	Test	PF 1,5	PF 2,5	PF 4,5	PF 7
Acetates	Hexyl acetate	240,9	199,6	161,5	100,0	86,6
	Isoamyl acetate	746,8	642,6	516,2	349,4	289,5
	Ethyl acetate	398,4	285,9	201,2	165,6	126,6
	SUM ACETATE	1386,1	1128,2	878,8	615,0	502,7
Ethyl esters	Ethyl butyrate (C4 ethyl)	55,7	44,8	35,8	23,6	20,1
	Ethyl caproate (C6 ethyl)	701,3	546,4	450,9	271,6	230,7
	Ethyl caprylate (C8 ethyl)	4762,3	3485,7	2815,9	1727,8	1136,4
	Ethyl Caprino (C10-ethyl)	4953,3	3846,5	2723,0	1701,3	743,7
	Ethyl-2-methyl-butanoate	1,0	0,8	0,6	0,5	0,4
	Ethyl-3-methyl-butanoate	0,8	0,8	0,6	0,5	0,4
	SUM ETHYL ESTERS	10474,5	7925,0	6026,9	3725,2	2131,8
C6 alcohols	Hexanol	168,7	172,2	116,2	108,2	72,1
	Trans-3-hexenol	4,3	5,0	3,4	3,0	2,1
	Cis-3-hexenol	28,4	32,4	17,7	19,0	13,6
	SUM ALCOHOLS C6	201,4	209,5	137,4	130,3	87,8
Alcohol various	Beta-Phenyl Ethyl Alcohol	12071,2	11242,7	6867,5	8560,2	6984,6
Terpene alcohols	Linalool	2895,3	2566,0	1885,4	2065,3	1707,3
	HO-Trienolo	262,5	354,3	172,5	476,6	528,2
	Alpha-Terpineol	329,8	472,1	208,7	627,7	688,1
	Citronellol after 1-Decanol	25,9	23,1	16,4	16,0	10,7
	Nerol under 4-Hydroxy Ethyl butyrate	36,4	44,9	25,0	55,4	52,4
	Geraniol ethyl below laurina	209,0	166,3	85,0	115,4	100,8
	D-Limonene	154,9	172,9	56,8	103,4	93,9
	SUM terpene alcohols	3913,7	3799,6	2449,9	3459,7	3181,4
Oxides of linalool	Trans-linalool oxide (C) (Pir)	24,4	29,0	20,2	30,6	29,2
	Cis-Rose oxide under hexanol	3,3	3,4	1,9	1,6	1,3
	Trans-Rose Oxide	0,7	0,6	0,4	0,2	0,2
	SUM linalool oxide	28,4	33,0	22,4	32,4	30,7
Terpene ethers	Linalyl Ethyl ether	479,0	534,8	160,3	249,5	172,6
	Neryl Ethyl ether	12,3	13,1	4,4	7,2	5,3
	Geranyl ethyl ether	259,3	275,6	15,0	6,0	449,8
	Alfa Terpenil Ethyl ether	12,4	14,4	10,5	15,7	14,8
	SUM ETHERS Terpenes	762,9	837,9	190,0	278,4	642,5
Vinylphenol	4-Vinyl-Guaiacol	40,7	28,4	26,1	29,5	26,3
	4 Vinyl Phenol	0,0	14,1	11,6	14,4	13,1
	SUM vinylphenol	40,7	42,5	37,7	43,9	39,4
Aromatic aldehydes	Benzaldehyde	12,1	34,0	19,0	30,3	25,2
Fatty acids	Butyric	15,7	18,2	12,7	14,9	12,6
	Caprylic acid (C 8)	2075,5	2262,0	1716,5	2185,6	2010,5
	SUM FATTY	2091,2	2280,3	1729,2	2200,5	2023,1
Norisoprenoids	Beta Damascenone SOT-dop Cinammato E	19,4	22,9	11,8	30,2	31,6
	Beta-Ionone	0,0	0,0	0,0	0,0	0,0

Tab. 2

		Sfurzat		Sfurzat		Chianti		Valpolicella	
COMPOUND CLASS	COMPOUNDS TESTED BY SPME	16,6 %v/v	-1,7 %v/v	15,9 %v/v	-1,3 %v/v	13,9 %v/v	-1,0 %v/v	14%v/v	-1,7 %v/v
Acetates	Hexyl acetate	4,3	3,8	13,6	14,5	8,8	5,1	21,4	12,6
	Isobutyl acetate	2,8	2,2	2,4	2,2	1,3	1,0	2,8	0,9
	Isoamyl acetate	79,4	71,5	161,9	147,1	107,6	83,3	339,6	204,0
	Beta-phenylethyl acetate	102,1	42,0	41,9	33,2	47,3	42,6	38,1	32,2
	Ethylphenyl acetate	7,3	5,4	4,5	4,5	4,9	4,8	2,4	2,2
	SUM ACETATE	196,0	124,9	224,4	201,5	169,8	136,7	404,3	251,9
Ethyl esters	Ethyl butyrate (C4 ethyl)	33,9	26,8	31,1	27,2	16,5	14,0	42,2	26,7
	Ethyl caproate (C6 ethyl)	327,7	294,6	350,3	320,7	240,9	222,1	493,0	342,0
	Ethyl caprylate (C8 ethyl)	1.400,1	862,2	1.070,3	945,9	1.018,3	1.345,8	2.163,6	1.639,5
	Ethyl Caprino (C10-ethyl)	783,5	343,3	537,5	431,2	731,6	946,4	742,8	513,0
	Ethyl-2-methyl-butanoate	6,6	5,9	6,1	5,2	5,6	5,7	1,7	1,2
	Ethyl-3-methyl-butanoate	10,5	9,1	8,9	7,8	6,8	6,0	2,9	2,1
	SUM ETHYL ESTERS	2.562,3	1.541,8	2.004,3	1.738,0	2.019,7	2.539,9	3.446,1	2.524,5
C6 alcohols	Hexanol	696,4	619,3	856,0	672,7	360,6	323,4	247,7	214,5
	Trans-3-hexenol	9,7	7,6	8,8	6,7	9,0	7,1	4,3	3,4
	Cis-3-hexenol	20,0	12,7	16,9	11,8	10,7	10,3	11,0	7,5
	2-Hexen-1-ol	1,4	1,3	1,5	1,0	0,9	0,7	0,6	0,4
	SUM ALCOHOLS C6	727,5	640,9	883,2	692,2	381,2	341,5	263,6	225,8
Various alcohols	Benzyl alcohol	49,8	37,6	33,1	22,8	57,4	55,6	13,7	11,5
	Beta-Phenyl Ethyl Alcohol	8.318,6	5.442,0	7.582,2	5.304,0	7.095,8	6.745,8	2.949,9	2.443,3
	3-Methyl-thio-propanol (Methionol)	36,8	28,5	42,2	25,1	64,0	69,2	28,6	23,7
	SUM VARIOUS ALCOHOLS	8405,2	5508,1	7657,5	5351,9	7.217,2	6870,6	2992,2	2478,5
Terpene alcohols	Linalool	32,8	32,4	28,9	24,4	44,5	42,4	141,0	155,2
	HO-Trienolo	4,1	2,2	1,8	1,6	0,4	0,3	0,7	0,7
	Alpha-Terpineol	18,4	15,5	13,1	10,5	9,8	9,0	34,4	38,2
	Citronellol after 1-Decanol	11,9	10,3	12,3	10,7	21,7	20,9	20,2	21,4
	Nerol under 4-Hydroxy Ethyl butyrate	3,9	2,6	3,9	3,1	3,0	3,5	4,3	4,8
	Geraniol ethyl below laurina	3,8	2,8	3,4	2,6	6,6	5,8	7,6	8,9
	D-Limonene	9,2	3,5	7,1	3,6	3,8	3,1	20,8	14,7
	4-Terpineol	103,3	89,8	143,7	115,7	2,8	2,7	2,0	1,8
	SUM terpene alcohols	187,5	159,0	214,2	172,3	92,7	87,6	231,0	245,8
Ethylphenol	4-Ethyl Phenol	18,6	14,1	13,3	10,8	19,8	59,8	3,8	24,3
	4 - Ethyl Guaiacol	4,7	3,6	5,7	4,6	5,9	17,5	1,0	21,1
Phenols varietal	Eugenol	7,2	5,0	3,3	3,0	1,5	1,4	3,6	3,8
	Guaiacol	4,8	3,5	3,3	2,3	0,9	0,8	1,3	1,2
	Ortho-Cresol	1,9	1,0	1,4	0,8	0,9	0,7	1,1	0,8
	Paracresol	2,7	1,5	1,9	1,5	0,8	1,0	1,0	0,7
	Phenol	7,6	4,7	6,8	4,7	3,4	2,9	7,9	6,6
	SUM PHENOLS VARIETY	24,2	15,7	16,7	12,4	7,5	6,9	14,9	13,2

		Sfurzat		Sfurzat		Chianti		Valpolicella	
COMPOUND CLASS	**COMPOUNDS TESTED BY SPME**	**16,6 %v/v**	**-1,7 %v/v**	**15,9 %v/v**	**-1,3 %v/v**	**13,9 %v/v**	**-1,0 %v/v**	**14%v/v**	**-1,7 %v/v**
Aromatic aldehydes	Phenyl Acetaldehyde	59,5	36,1	33,2	21,9	26,0	23,3	17,2	11,8
	Benzaldehyde	135,2	115,5	117,7	91,3	39,2	17,9	31,9	10,4
	SUM aromatic aldehydes	194,6	151,6	150,9	113,2	65,2	41,2	49,1	22,2
Oak Lactones	Oak Lattone 1 (Whiskey lactone)	30,5	23,0	11,9	9,4	2,2	2,1	0,8	0,0
	Oak Lattone 2 (Whiskey lactone)	51,6	38,9	23,6	19,5	2,6	2,8	0,0	0,0
	SUM OAK Latton	82,1	62,0	35,5	29,0	4,8	4,9	0,8	0,0
Fatty acids	Butyric	21,2	15,1	21,0	14,6	9,5	8,6	20,9	18,3
	Ac Iso-Valeriani	33,1	27,1	34,1	27,0	53,4	51,6	34,4	30,5
	Caproic acid (C 6)	316,6	234,3	276,6	205,0	160,0	165,9	275,8	259,9
	Caprylic acid (C 8)	1.009,5	629,3	665,0	527,6	543,5	505,1	835,8	896,0
	Capric acid (C 10)	408,6	208,8	229,8	186,7	484,4	273,7	242,6	269,3
	SUM free fatty	1.789,0	1.114,6	1.226,6	960,9	1.250,9	1.004,9	1.409,5	1.474,0
Norisoprenolds	Beta Damascenone SOT-dop Cinammato E	12,0	10,2	19,5	17,1	15,6	14,5	36,9	39,2
	Alpha-Ionone	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
	Beta-Ionone	1,0	0,8	0,8	0,5	0,5	0,5	0,5	0,4
	TDN	50,9	23,6	50,5	29,5	12,4	9,9	10,6	7,6
	Ethoxy TDN	2,5	2,1	2,4	2,3	1,2	1,5	1,1	1,3
	Vitispirano 1	305,1	278,0	341,7	265,1	48,3	48,9	86,3	77,9
	Vitispirano 2	103,1	91,3	115,0	94,4	25,3	25,6	57,7	49,1
	Actinidoli Ethoxy Derivatives 1	17,9	15,8	14,2	12,8	3,8	4,0	1,6	2,1
	Actinidoli Ethoxy Derivatives 2	11,4	9,8	8,5	8,0	2,5	2,5	1,1	1,4
	Actinidoli 1	3,3	2,6	2,4	1,9	0,9	1,2	0,6	0,7
	Actinidoli 2	5,5	3,8	4,5	3,7	2,0	2,1	1,2	1,3
	SUM NORISOPRENOIDS	512,6	437,8	559,6	435,1	112,4	110,8	197,6	181,1

Tab. 3



INTERVITIS INTERFRUCTA 2010

International technology trade fair for wine, fruit, fruit juice and spirits

Meny years' experiences on the dealcoholization processes using membrane techniques to guarantee the quality and sustainability of the wine

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Why to dealcoholize?

- Social reasons
- Health reasons
- Due to climate change

Climatic changes and the increasing of alcohol content in wines: a global emergency

- The Australian Wine Research Institute found that the average content of alcohol in Australian wines increase from 12.4% to 14% from 1984 to 2004 (average of 15% Shiraz)
- United States ---------->
- Similar trend for other producer countries

(2) ALCOHOL & SUGAR LEVELS IN CALIFORNIA
From CA Grape Crush Report District 4 – All varieties

Year	Alcohol
1971	12.5%
1973	12.7%
1975	13.2%
1976	13.7%
1978	14.0%
1981	13.7%
1983	13.1%
1986	13.3%
1990	13.5%
1992	13.8%
1997	14.4%
1999	14.6%
2001	14.8%

Extracted from Conibear (2006)

Reduction of the wines alcohol content

- Production of grapes with lower sugar content, but ”ripe"
  - Agronomic practices
    - Increase the yield
    - Still need to produce ripe and "balanced" grapes
  - Direct action on the grape-vine (selection, GMOs ...)
- Reduction of alcohol yield during the alcoholic fermentation
  - Low alcohol producers yeasts
- Changing the composition of musts or wines (decrease the concentration of sugars or alcohol)
  - Dilute the musts (and wine treated with RO)
  - Reduce sugar content of musts
  - Wine dealcoholization

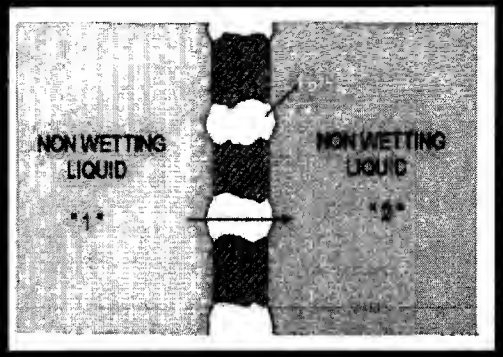
Wine dealcoholization methods

- Physical techniques directly on wine: Spinning Cone Column, runoff evaporation at atmospheric pressure, vacuum evaporation, supercritical extraction of alcohol with CO_2, other physical techniques.
- Membrane processes in combination with other techniques

Membrane technologies

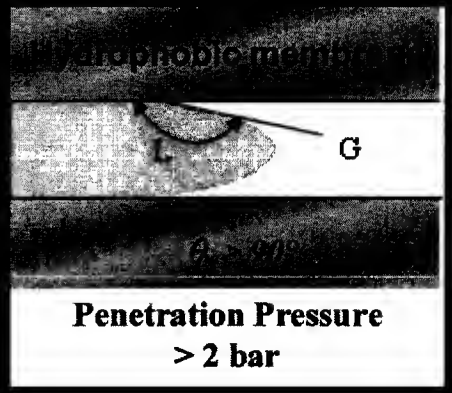
- RO + Distillation
- RO + Water
- ***"Contactor" membrane***
- ***Combination of membrane techniques***
- ***Pervaporation***

Gasseous Contactor Membrane



Principle

- *Hydrophobic membranes*
- *Operating P < Penetration P*
- *Makes contact between two liquids keeping them separated by a layer of gas*



Applications

- *Osmotic Distillation*
- ***Separation by gasseous membrane***

Contactors application in wines treatment: decreasing alcohol content (gas membrane separation)

- Driving force is a Δ in vapour pressure created by a Δ in concentration
- Extractant: water

$$N_{eth} = K_{meth} \cdot \frac{P_{eth\,1} - P_{eth\,2}}{P_{1\,atl}}$$

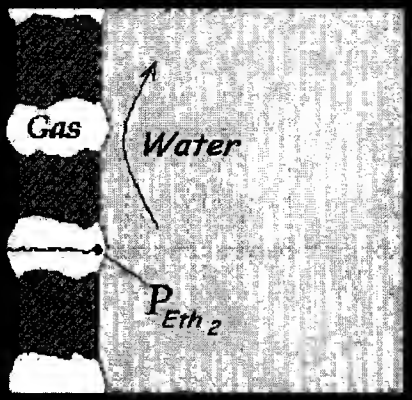
N_{eth} = *ethanol molar flow*

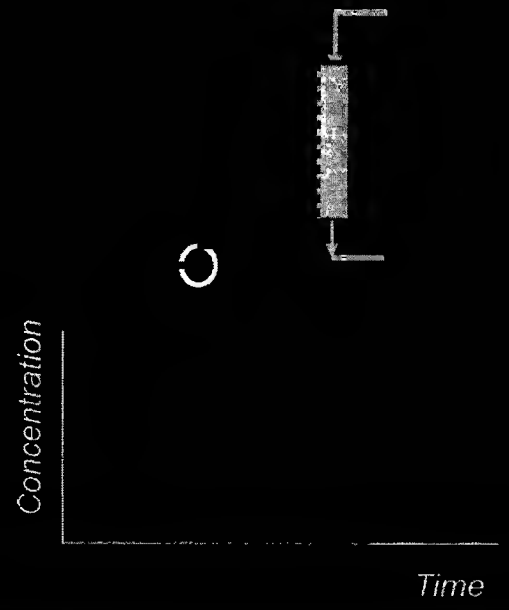
P_{eth1} = *ethanol vapour pressure in the wine*

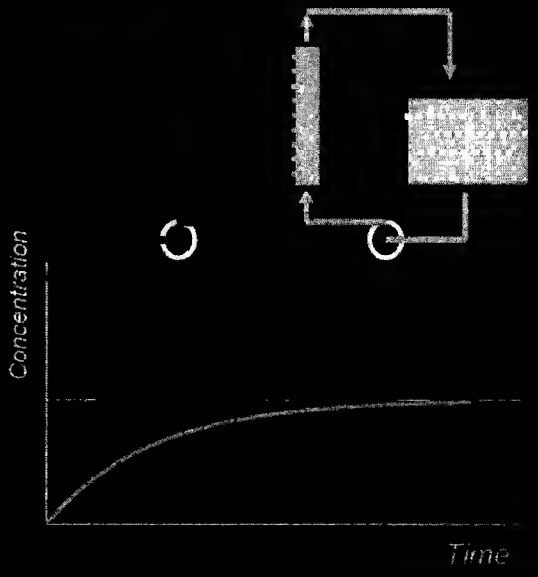
P_{eth2} = *ethanol vapour pressure in the extractant*

K_{meth} = *Diffusional permeability* = $K_{mw} D_{eth}/D_{W,1}$

Alcohol reduction on wine

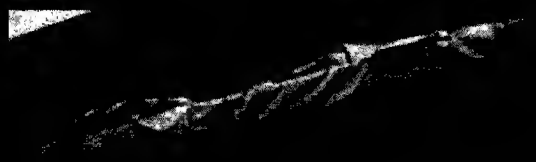




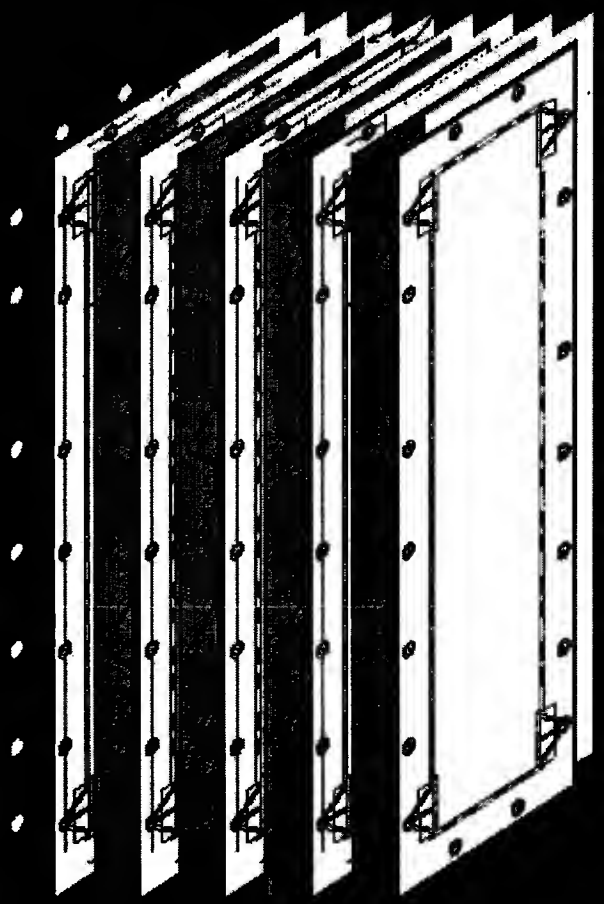


Contactor Membrane

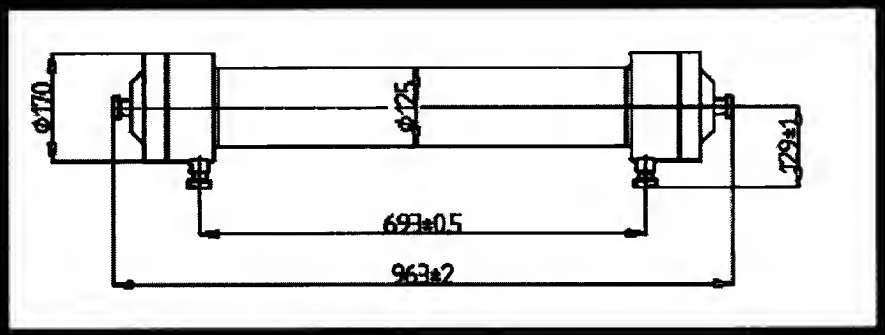
PILOT PLANT



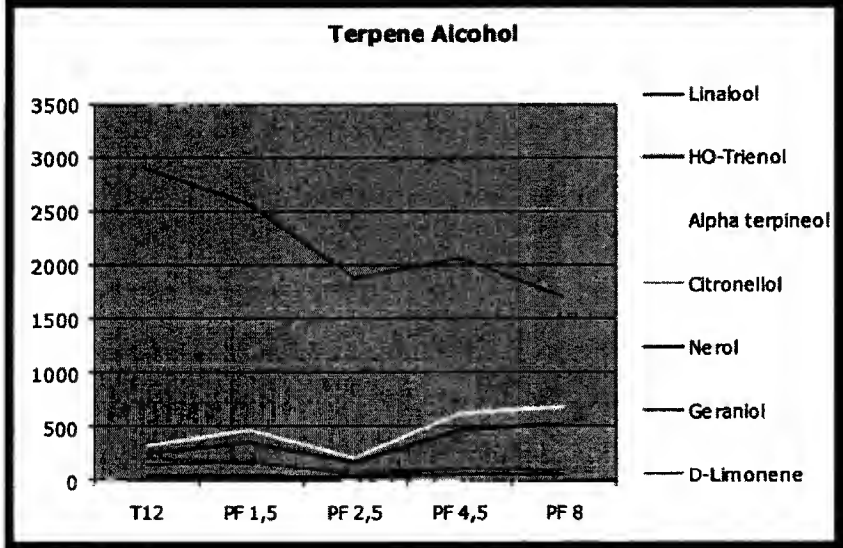
FRAME AND PLATE

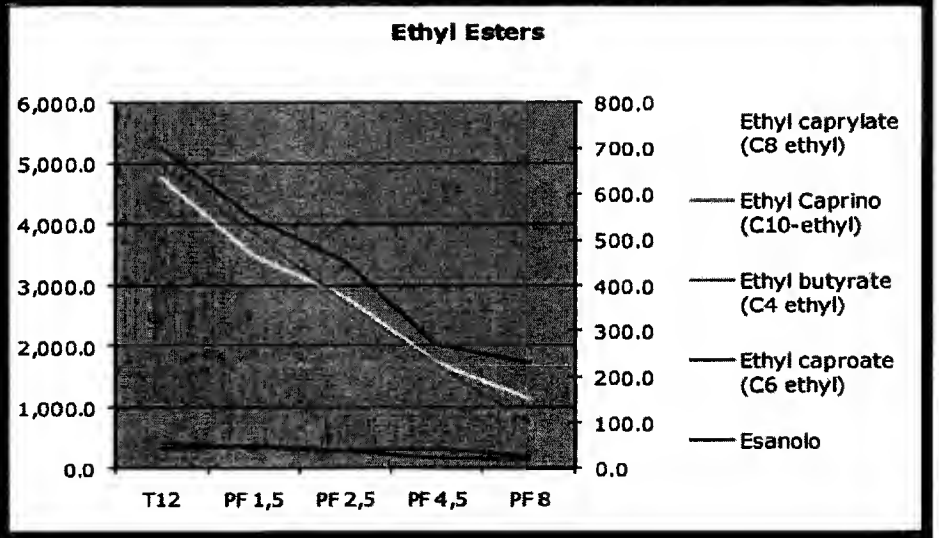


HOLLOW FIBER

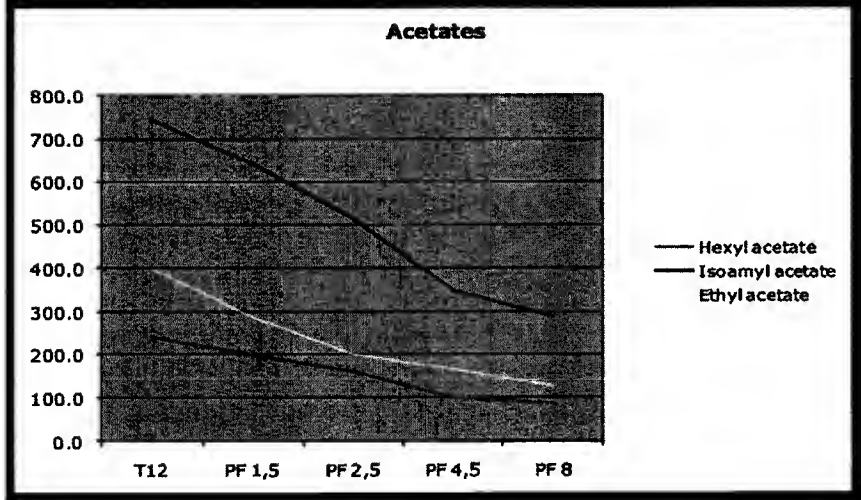


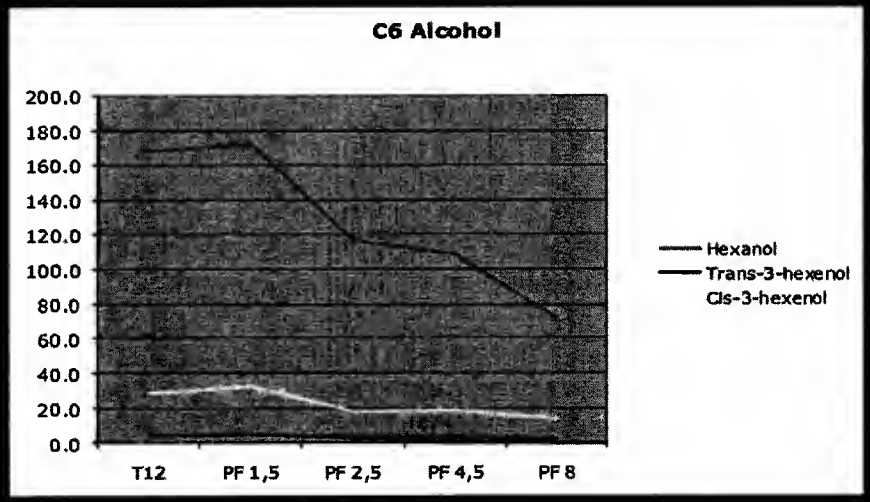
Study of the effects of the dealcoholization rate with contactor membrane on the wine composition





Study of the effects of the dealcoholization rate with contactor membrane on the wine composition

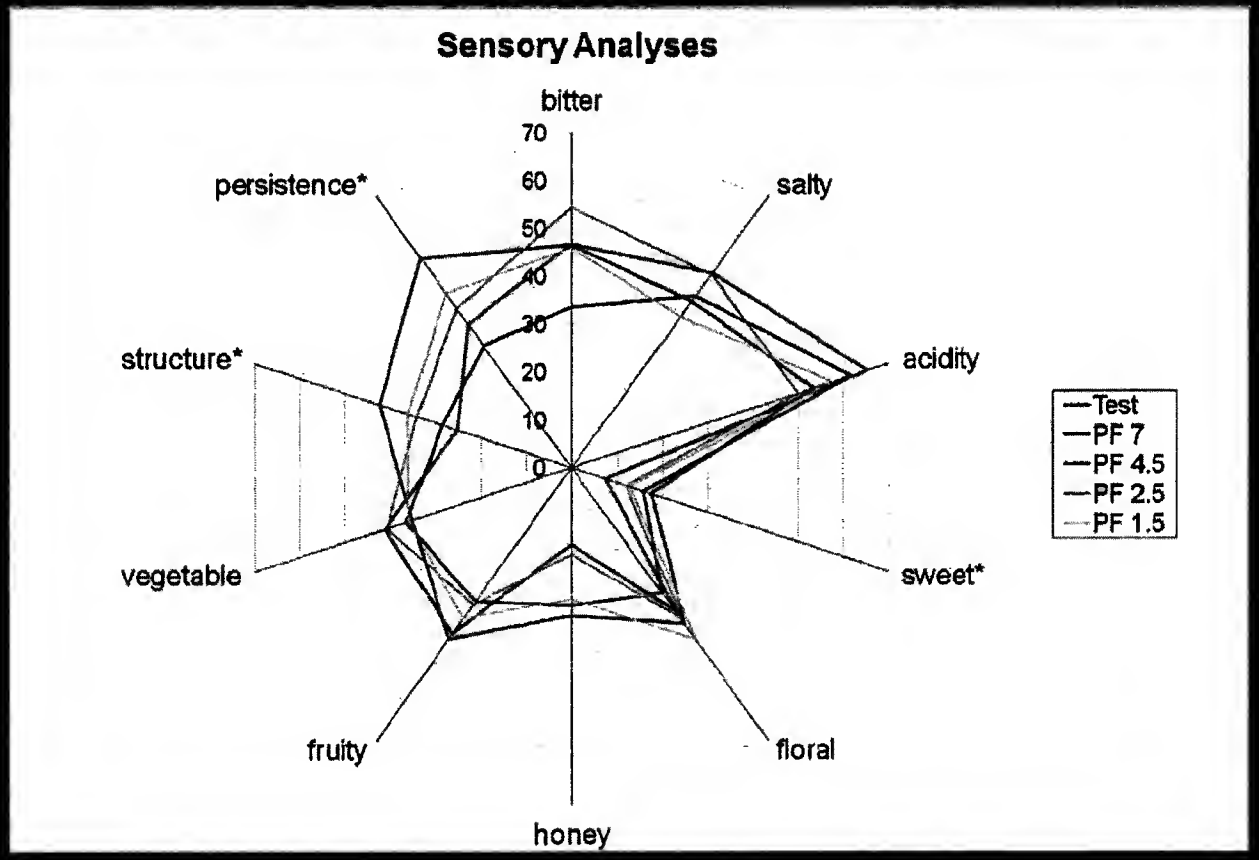




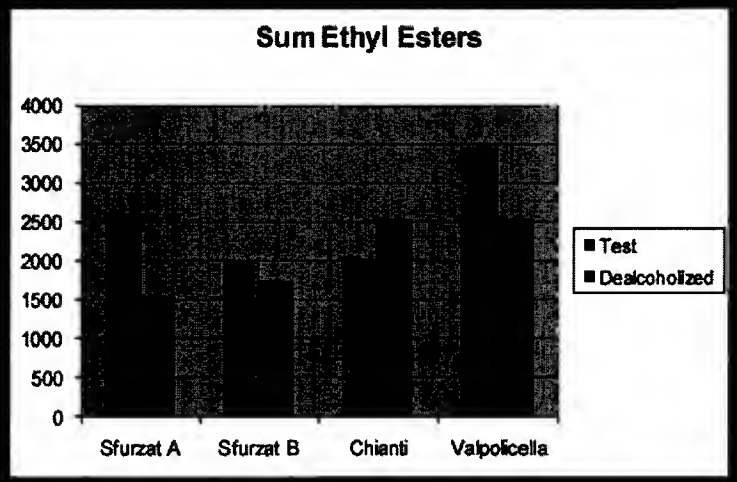
Study of the effects of the dealcoholization rate with contactor membrane on the wine composition

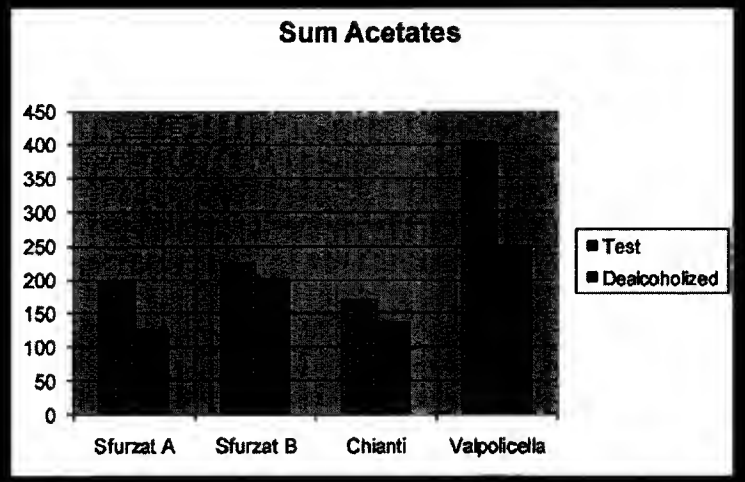
% Alcohol removed		0 Test	1,4	2,7	5,1	7,1
Alcohol	(% vol)	12,4	11,0	9,7	7,3	5,3
pH		3,46	3,44	3,49	3,48	3,47
Titratable Acidity	(g/L)	6,6	6,6	6,7	6,7	6,9
Volatile Acidity	(g/L)	0,07	0,06	0,06	0,05	0,05
Acetic Acid	(g/L)	0,01	0,01	0,01	0,01	0,01
Total exctract	(g/L)	23,5	23,9	23,2	24,2	24,7
Glycerol	(g/L)	10,1	10,8	10,8	11,1	11,5
Tartaric acid	(g/L)	1,82	1,79	1,90	1,88	1,89
K	(mg/L)	638	664	653	675	709
Ash	(g/L)	1,89	1,80	1,81	1,81	2,17
Ash alkalinity	(meq/L)	20,23	18,8	19,0	21,0	21,3
Saturation temperature	(°C)	22,1	20,4	20,1	17,8	16,5

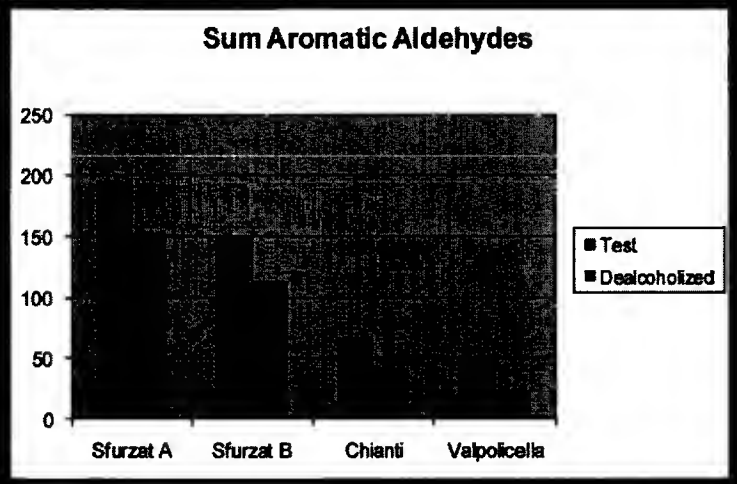
Sensory profile of dealcoholized Moscato

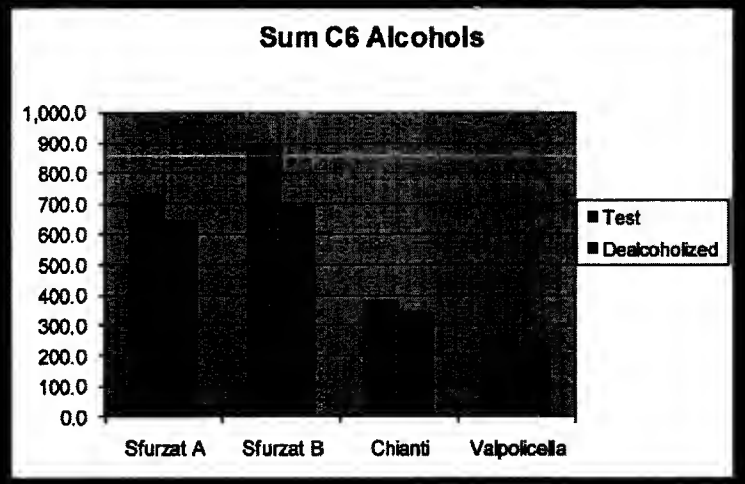


Industrial low dealcoholization (up to 2%) experiences on typical Italian wines

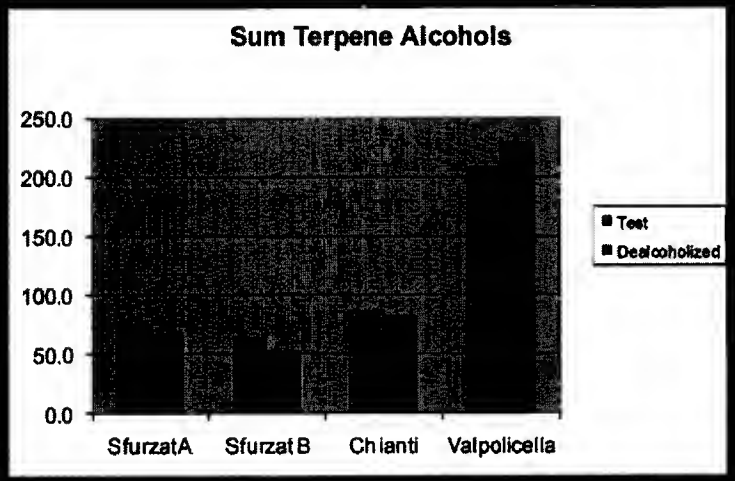


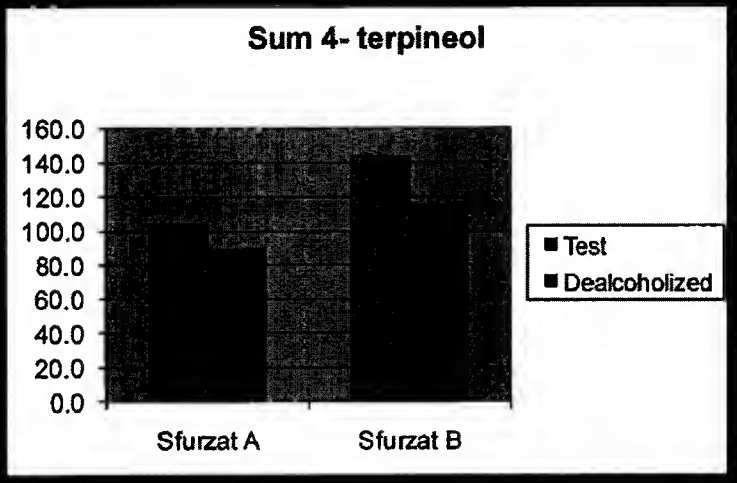


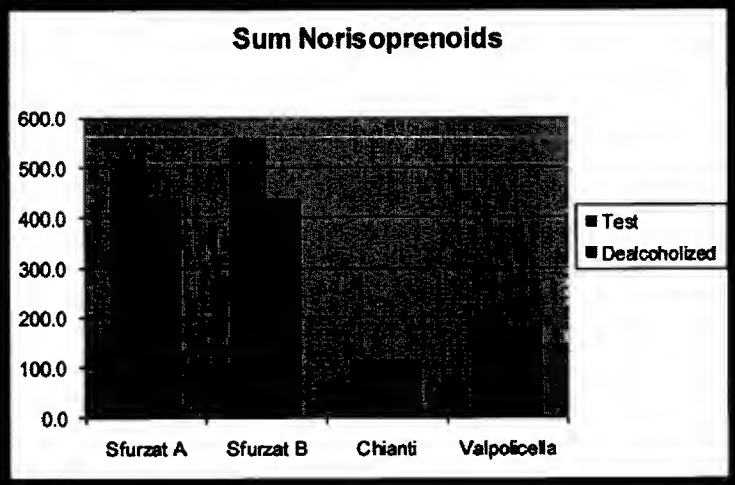


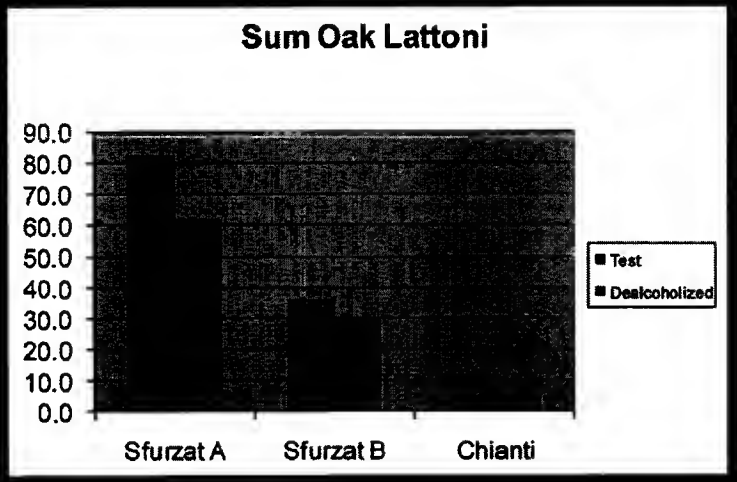


Industrial low dealcoholization (up to 2%) experiences on typical Italian wines

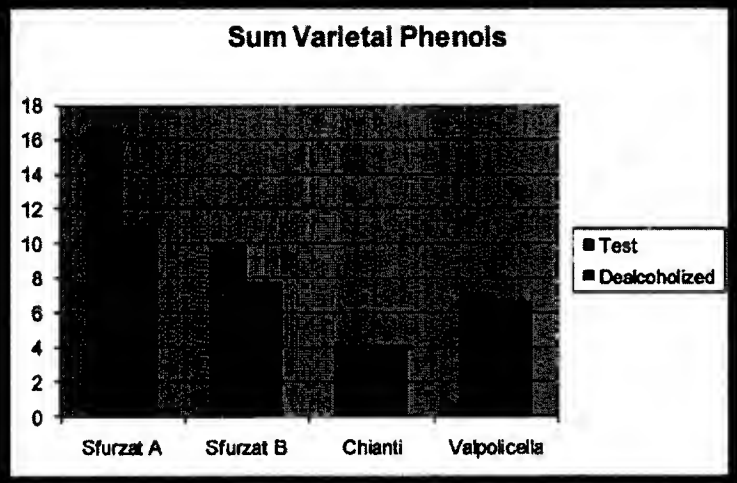


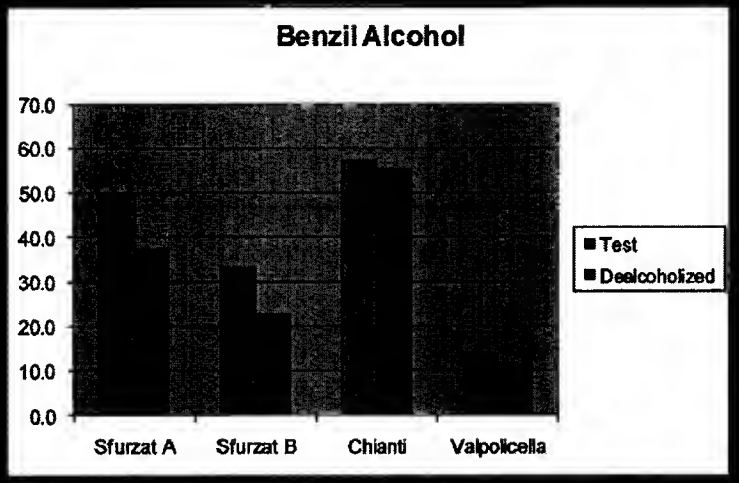


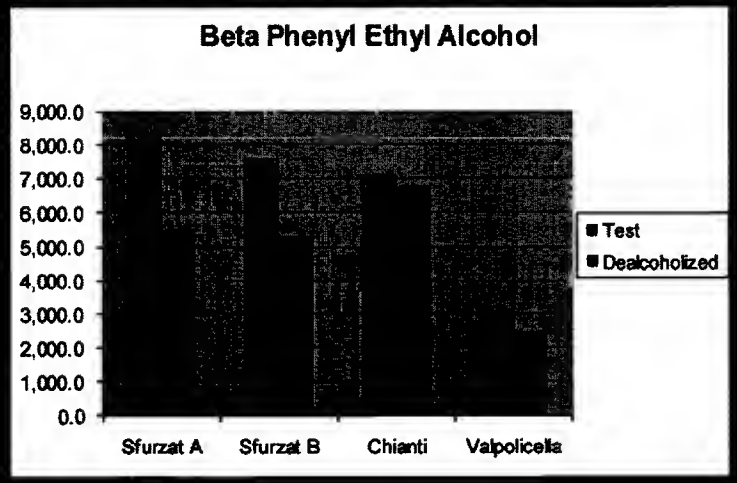


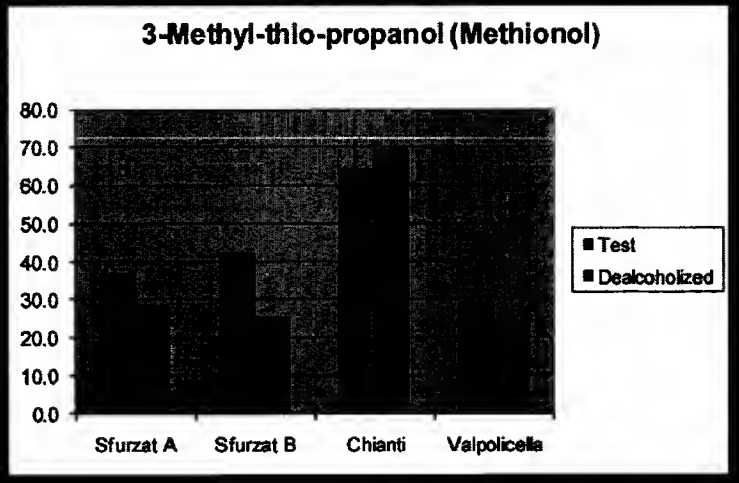


Industrial low dealcoholization (up to 2%) experiences on typical Italian wines

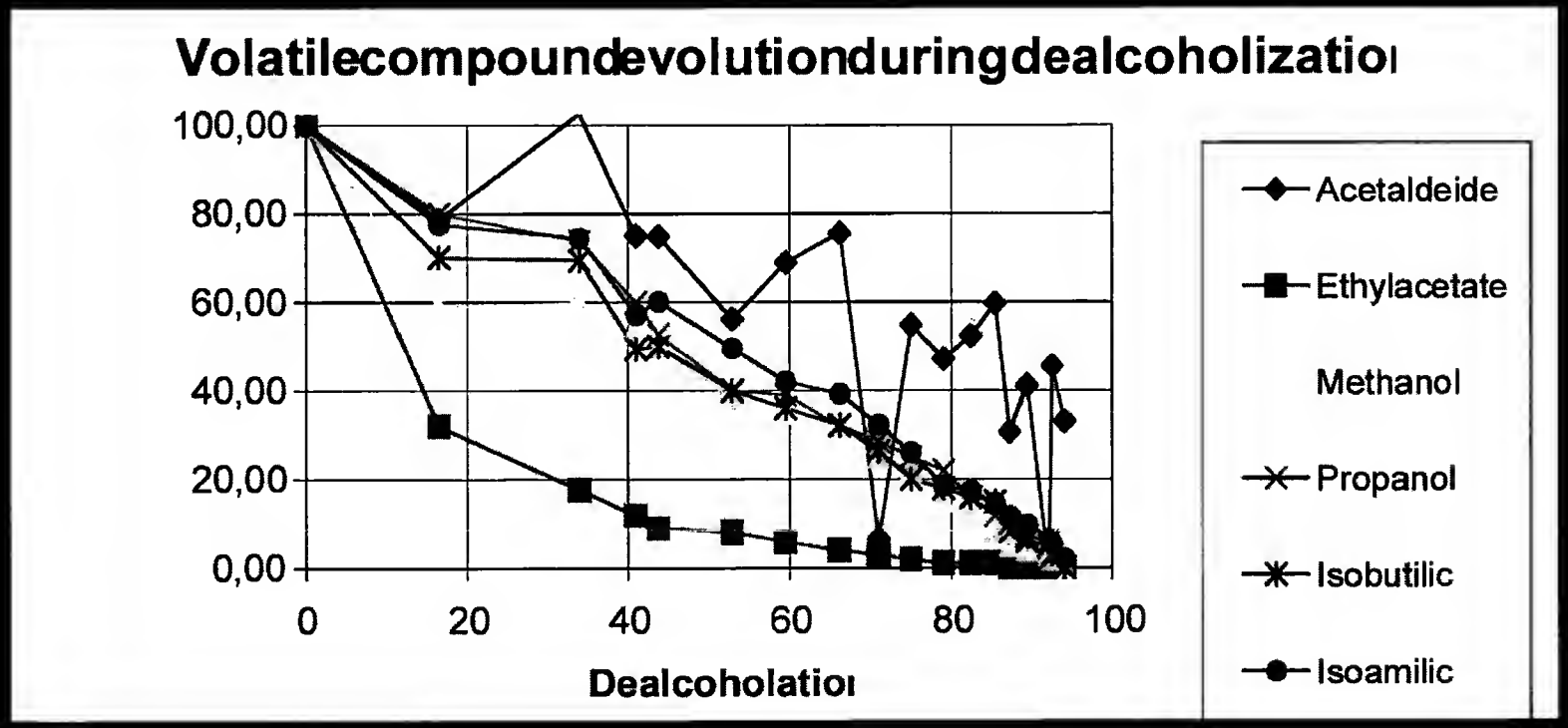








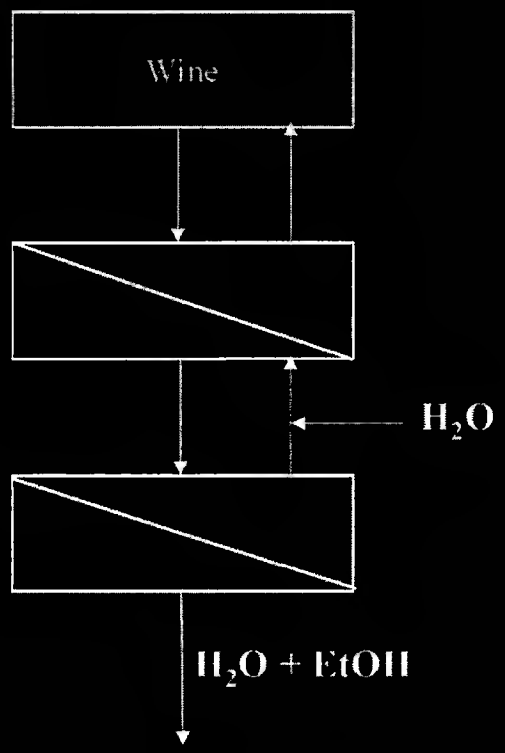
Other volatile compounds removed during dealcoholization process

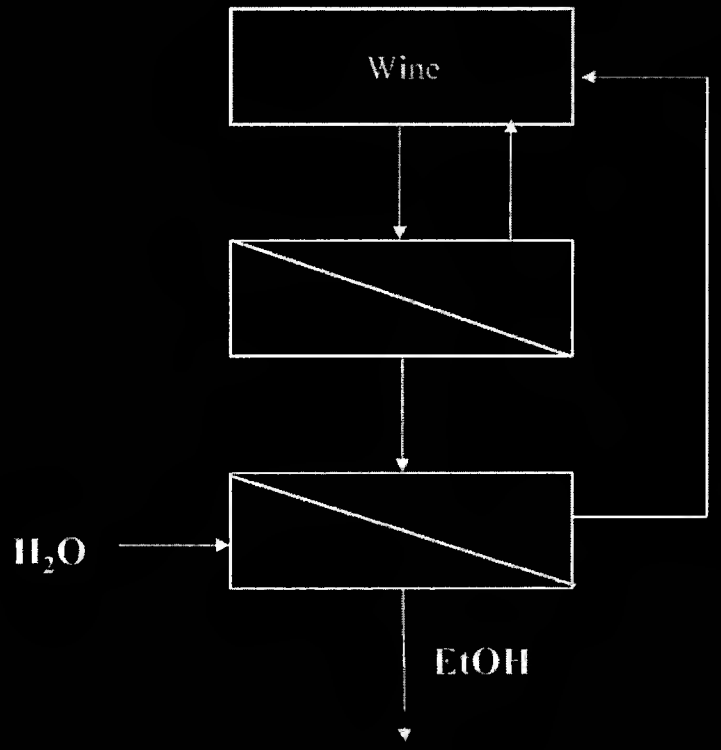


OTHER SCHEMES FOR DEALCOHOLIZATION COMBINED WITH MEMBRANE

1

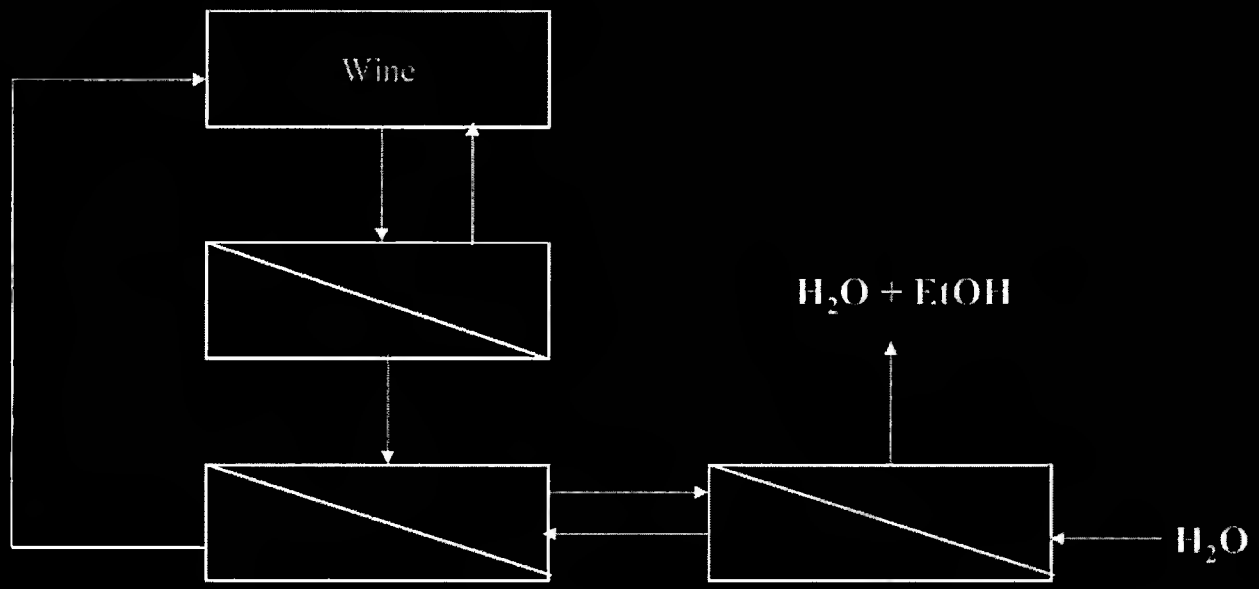
TO AVOID LOSS OF FLAVOR





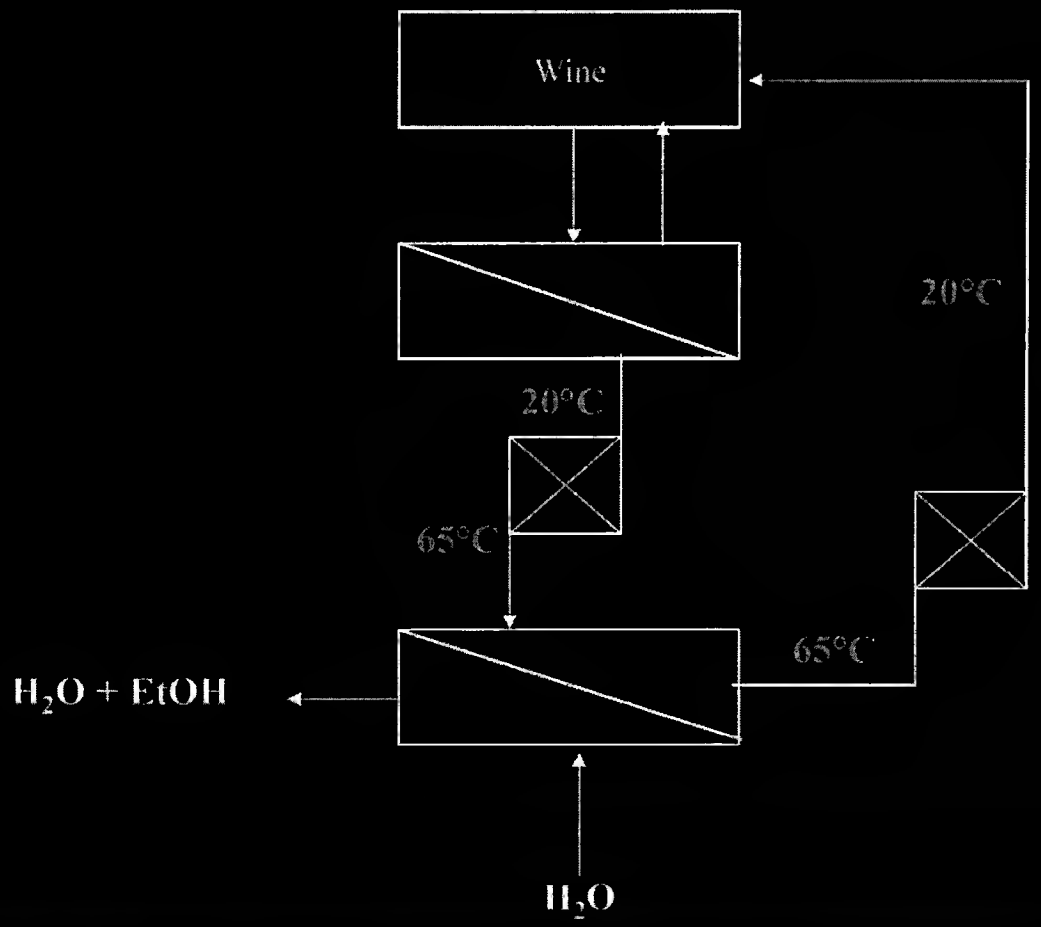
2

TO REDUCE THE CONSUME OF WATER



3

TO IMPROVE YIELDS



Results and considerations about "membrane contactor" techniques

- This technology allows a partial dealcoholization with quality results similar to other techniques.
- Compared to other techniques currently evaluated by OIV, it permits partial wine dealcoholization with only one treatment and without splitting the wine.
- This technique, in conjuction with anionic resins, can also be used to remove some other volatile compounds from the wine, for example acetic acid: however the treatment doesn't require wine splitting because the acetic acid elimination occurs in the external environment of the product (extractant solution).

Results

- To communicate the dealcoholization on the label gives an higher value to the wine for the costumer
- There are contraddictory effects concerning the sensory profile (mainly with strong dealcoholizations ...)
- Other studies of the same authors highlights the complexity of the dealcoholization effect and its relation with the type of wine, the tratment rate...

When to deacoholize?

- To re equilibrate a wine
- To produce a low alcohol content wine, always respecting the minimal alcohol content required by law: it's necessary to understand which is the lowest alcohol content that still characterize a wine under the sensory and cultural profiles
- To produce new beverages from grapes

Conclusions

- Dealcoholization, more than an opportunity, is often a necessity also in the production of a “tradictional” wine
- Certainly dealcoholization can be an opportunity considering the present purchases junction and the social needings concerning the alcohol intake
- The technique is interesting on te “Prosecco”…

Acknowlegments

- Vason Group / JU.CLA.S. - Italy
- Prof. Carlo Gostoli – Chemical Engineering Department – University of Bologna (Italy)
- Collaboratori DiSTeMeV: C. Carbognin, E. Nicolis, E.M. Casarotti, M. Manzo, N. Righetti

PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.:	10/563,636
Filing Date:	March 14, 2008
Confirmation No.:	3139
Applicant:	David Wollan
Group Art Unit:	1781
Examiner:	Vera Stulii
Title:	Alcohol Reduction in Beverages
Attorney Docket:	3029-000089/US/NP

Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

SECOND DECLARATION OF DAVID WOLLAN UNDER 37 C.F.R. § 1.132

I, David Wollan, declare:

1. I am the inventor named in this patent application.

2. I have worked as a winemaker and wine technologist for over 30 years. Since 1997, I have been involved in the application of membrane based techniques in winemaking - in particular, the use of reverse osmosis and nanofiltration as part of a membrane separation, treatment and recombination strategy for manipulating wine composition.

I have regularly been invited to be a presenter on wine technology matters at local and international oenological conferences. Most recently, together with other experts, I was invited to offer two presentations on alcohol management and technological options for this at the 2010 Intervitis Interfructa International Congress in Stuttgart, Germany. (Copies of my presentations which explain and compare the various technologies for alcohol adjustment are attached.)

Over the years I have also been a regular contributor to wine industry and oenological publications. Most significantly, I contributed a chapter: "Membrane and other techniques for the management of wine composition" in Reynolds, AG (Ed.) "Managing wine quality, Volume 2: Oenology and wine quality", Woodhead Publishing Ltd, 2010. (http://www.woodheadpublishing.com/en/book.aspx?bookID=2050)

In 2006, the organization Australian Wine Selectors named me as "Wine industry innovator of the year" for a range of innovations including the claimed process of this application.

3. I am familiar with the methods that were used prior to my invention to reduce alcohol content in beverages, in particular in wine. I have read and understood the Michaels publication and the Nielsen publication, and I am familiar with the processes they describe.

4. Pressure-driven separation of alcohol from water solution by itself does not work satisfactorily. The Nielsen process would not work in the way Nielsen describes, as evidenced by Michaels et al., U.S. Patent 5,817,359, filed shortly after publication of the Nielsen document. In column 1, line 53 to column 2, line 5, Michaels criticizes dealcoholization by reverse osmosis (the method used in the Nielsen process) because desirable wine components are lost because

they are passed into the permeate which is discarded, and the rententate (wine) is diluted with water:

> Dealcoholization by reverse osmosis removes from the beverage not only volatile low molecular weight components such as water, alcohol, flavor and fragrance components, and dissolved gases such as carbon dioxide and sulfur dioxide, but may also remove certain low-molecular-weight, nonvolatile, nonionic solutes such as organic acids and simple sugars. As a consequence, removal of a significant amount of alcohol from a beverage by reverse osmosis may be accompanied by the removal of proportionately large amounts of these other membrane-permeable components. Current practice involves dilution of the reverse-osmosis concentrate or retentate with pure water to replace the water removed in the permeate. As a result, the final dealcoholized product is substantially depleted in other membrane-permeable solutes, with resulting undesirable changes in flavor, fragrance, color, acidity, stability, and the like. Restoration of these depleted components by their re-addition to the concentrate in proper proportions to produce a high-quality product is generally difficult, costly, or impossible.

The reverse osmosis step of the Nielsen process (described on page 8, lines 10-23, page 11, line 34 to page 12, line 9, page 13, lines 5-13, page 18, lines 25-35, and page 22, lines 25-33) cannot achieve satisfactory pressure-driven separation of alcohol from water solution because, if typical reverse osmosis membranes are employed as in Nielsen, the separation as described is by the molecular weight of the various species, e.g., water and ethanol. This means that, in general, water with a molecular weight of 18 will pass through all common membranes at a greater rate than alcohol with a molecular weight of 44. So relative to the feed streams described by Nielsen, the permeate stream will be lower in alcohol concentration because of this greater rate of water passage and, logically, the retentate containing most of the desirable wine components will be *higher* in alcohol relative to water after the reverse osmosis step. Nielsen's whole process is based on the proposition that the reverse osmosis membrane will retain the high molecular

weight flavours (>100) and low molecular weight water (18), but somehow preferentially pass the in-between molecular weight alcohol (MW=44), which is contrary to the principles on which reverse osmosis works.

Because reverse osmosis works on these principles, when Nielsen's retentates of the microfiltration, nanofiltration, and reverse osmosis stages are eventually recombined, as they are in all of Nielsen's embodiments, the resulting recombination must be a beverage higher in alcohol concentration. This is borne out by the experimental data I provide in the Experimental Section, which follows below.

5. Notwithstanding the inconsistency between what the Nielsen document says happens during its reverse osmosis step and what must (and does) happen because of the principles by which reverse osmosis processes operate, even if it were possible to use a reverse osmosis membrane to separate water preferentially from an alcohol solution, which as just discussed cannot be done under the conditions described in the Nielsen document, fundamental thermodynamics make this practically impossible for the Nielsen beverages. If there were a reverse osmosis membrane that was truly selectively permeable to water molecules but retained the larger alcohol molecules, the osmotic pressure of a 25% by volume alcohol solution would be about 100 atmospheres. This means that even if a reasonable flux (which is defined as the flow of permeate through a unit of membrane surface area per unit time) could be achieved, to separate water from a concentrated alcohol solution would require huge amounts of energy. To explain, let's say we wish to concentrate a 10% by volume alcohol solution to a 25% solution as above, using an "ideal" reverse osmosis membrane which preferentially passed water but little, if any alcohol:

For every 1000 litres of 10% feed, there would be generated 400 litres of 25% alcohol retentate and about 600 litres of water permeate.

As I show below in the Experimental Section, the membrane with the best performance for separation of alcohol from water was the Vinocon RO-3, which had a permeate flux of about 5 litres per metre squared per hour. Therefore, to generate 600 litres per hour of water permeate would require at least 120 m^2 of membrane area.

Now using Nielsen's preferred figures for feed flow rates of 20-40 $l/m^2/h$, this would require a total flow rate of 2,400 to 4,800 litres per hour, all of which would need to be pressurised to in excess of 100 atmospheres (~10,000 kPa). The pump power required to do this according to various suppliers would be from 5 to 15kW.

Not only would a large amount of energy be required for what is, in effect, an incomplete separation (for every litre of alcohol removed, so also is about 3 litres of water or, effectively wine), but it would require unrealistic and impractical engineering specifications. Few pumps or associated fittings with material specifications suitable for treating beverages are able to generate pressures as high as 100 atmospheres; nor are there membranes or housings which can tolerate such high pressures.

In sum, the Nielsen method of using reverse osmosis as disclosed in that document will not result in a permeate relatively higher in alcohol, but instead must, by the principles of reverse osmosis, do just the opposite—concentrate alcohol in the retentate. Even if there were a reverse osmosis membrane that did retain the relatively smaller water molecules but pass the larger ethanol molecules, the laws of thermodynamics would make such a process unworkable.

6. The Michaels process for dealcoholisation of wine (also called "osmotic distillation" or "evaporative perstraction") was recognized as unsuccessful, as disclosed in published material from Michaels himself because of unacceptable flavour loss. This problem is admitted by Michaels himself in Michaels, U.S. Patent 5,817,359, column 2, lines 39-52, where Michaels candidly admits about the process in his earlier patent document,

> However, isothermal membrane perstraction has serious limitations which can have adversely affect the properties, characteristics, and quality of a dealcoholized beverage obtained thereby. Since the membrane employed for that process is freely permeable to any volatile solute which may be present in either liquid phase in contact with it, the volatile components present in the original beverage are free to leave the beverage and dissolve in the strip solution if the activity of any such solute is higher in the beverage than in the strip solution. Thus, volatile flavor and fragrance components, as well as dissolved gases such as carbon dioxide and sulfur dioxide, which may be present in the original beverage, can be depleted during the perstraction process.

In this passage, Michaels admits there is an issue with the free movement of volatile substances (flavor loss) from the wine into the strip solution. The preferred embodiments suggested to overcome this involve the recycling in the strip of some previously processed alcoholic strip or dealcoholised product. This necessarily results in the loss of product or the reduction in the rate of alcohol transfer as a result of lower differential alcohol concentration across the membrane.

These problems are recognized generally in the industry, as illustrated by a paper by Dr Ferrarini (attached) that was presented by E Bocca, a representative of Vason Group, at the 2010 Intervitis Interfructa International Congress in Stuttgart, Germany, . The Ferrarini document (slides 15-18) shows significant losses of a range of key flavour compounds for typical 2% dealcoholisation.

Because of the flavor loss problem, to the best of my knowledge, the process has not achieved significant industry acceptance, notwithstanding that there is a clear commercial need.

The Michaels document WO 93/22036, on page 9, acknowledges another serious problem with its process, the tendency for water to pass from the strip solution across the porous membrane into the wine. This was very undesirable as in most jurisdictions the addition of extraneous water to wine is prohibited. By way of example, in the Federal Register / Vol. 69, No. 223 / Friday, November 19, 2004 / Rules and Regulations pp 67642 – 67643, the US Department of Treasury Alcohol and Tobacco Trade and Tax Bureau [TTB] proposes to authorize the process (called here "Osmotic Transport") subject to certain conditions, including:

> *"The treatment must not alter the vinous character of the wine. The stripping solution must not migrate into the wine."*

Had the TTB been aware of the experimental evidence in the Michaels documents of water flux from the stripping solution into the wine, it may not have been prepared to authorize the process. Michaels proposed to overcome these limitations of his invention by manipulating the composition of the extracting solution. In my opinion this would inherently reduce the practicality and performance of the technology.

7. I discovered that an unexpected benefit in the preliminary step of creating a permeate stream that is dealcoholised instead of the whole wine (as in Michaels) is that the osmotic pressures of the solutions on both sides of the hydrophobic membrane become more equalized. This effectively overcomes any tendency for water flux into the permeate and hence the wine. This is an unexpected improvement compared to prior art processes, on top of the flavour preservation benefits. My invention provides these results that are different in kind and degree

than those that were available using the prior art processes described in the Nielsen and Michaels publications.

8. The embodiment in my claims 7, 8, and 36-38 offers a further unexpected benefit being able to carry out the alcohol reduction (perstraction) step at elevated temperature without damage to the wine product. This also could not be foreseen from the prior art processes, because the perstraction process is, by its inherent nature, isothermal i.e. energy is not required to drive the migration of alcohol vapor across the hydrophobic membrane. The alcohol vaporization and condensation process is driven by the alcohol concentration gradient across the membrane - no heat is gained or lost. What could not have been appreciated from the Nielsen and Michaels documents is that the kinetics of this process would be greatly enhanced at higher temperatures (subject to the limits tolerated by the membranes). This is a result of a combination of factors including enhanced vapor diffusion rates, reduced surface tension and reduced viscosity. In this regard, the proposal of Zhang to use heat to enhance mass transport of non-volatile phenolic materials across a non-porous membrane relies only on the process of solid diffusion and as such is only superficially similar to our observed phenomenon.

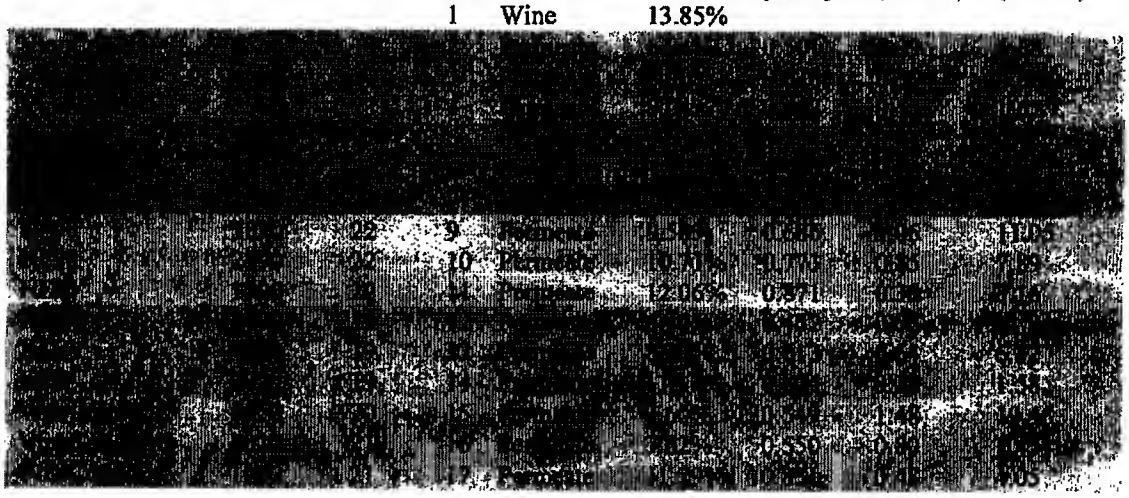
9. Experimental Section

In 2003 as part of our research and development program, I tested the performance of a number of commercially available reverse osmosis and nanofiltration membranes. Amongst other measures, I was looking for the total permeate flux at different pressures as well as the relative flux of alcohol. These figures gave an indication of the "passage" of alcohol which is defined as the ratio of alcohol concentration in the permeate to the alcohol concentration in the

wine feed. It is the inverse of "rejection". In all cases, the alcohol concentration in the permeate is lower than in the wine. In other words, water passes through the membranes more readily than alcohol. In no case was there less than 50% alcohol passage (>50% rejection).

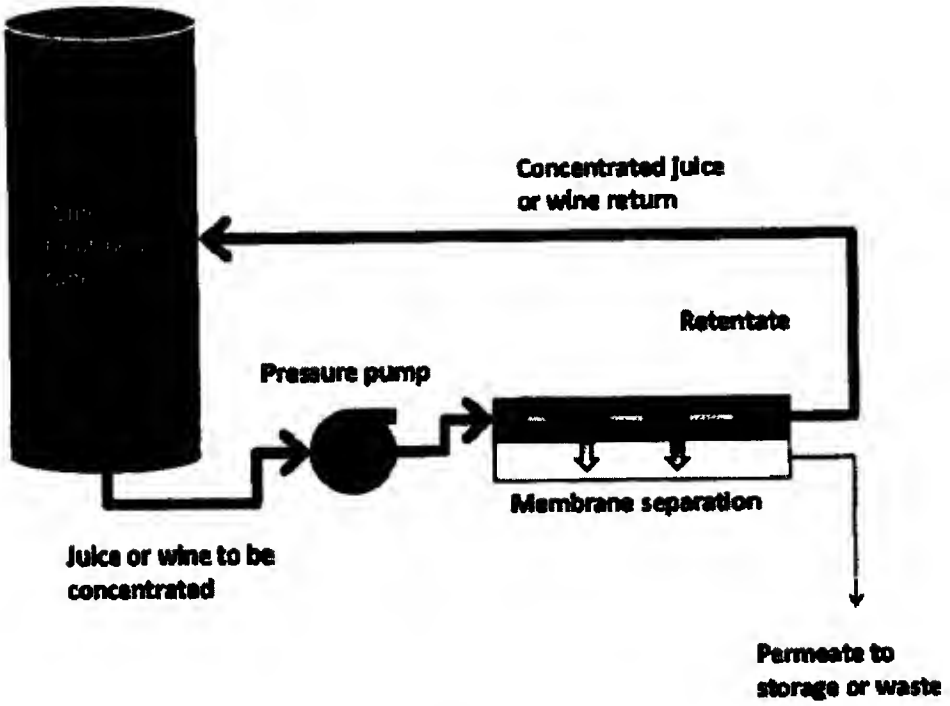
The Vinocon RO-3 and RO-5 as well as the XLE would be considered reverse osmosis elements (Nielsen element 35), with Vinocon RO-3 having the best water/alcohol separation characteristics. The VinoPro and NF90 are typical nanofiltration membranes (Nielsen element 24). It should be noted that in all instances, the alcohol concentration in the permeate is lower than in the feed and, as a corollary, in the retentate. This is clearly contrary to Nielsen's claims of what happens in the Nielsen process.

Membrane type	Pressure kPa	Temp °C	Sample#	Alcohol %v/v	Alc passage	Alc flux ($l/m^2/hr$)	Total Flux ($l/m^2/hr$)
			1 Wine	13.85%			



On another occasion, I tested the alcohol and acid passage performance, comparing the wine concentration effects of two different membrane types.

In these trials a volume of wine was concentrated as shown below such that 8% of the volume was separated as permeate.



That permeate, and the remaining retentate or concentrate (comprising 92% of the original volume), were analyzed for a couple of key parameters – titratable acidity and alcohol. This was repeated for NF and RO type membranes and the results illustrate the different permeabilities of these with regards to wine acids and alcohol.

	Titratable Acidity (g/l as tartaric acid)	Alcohol (% v/v)
Control wine (feed)	**5.9**	**14.10%**
8% Concentration with "loose", NF type membranes:-		
Permeate	**2.4**	**13.35%**
Retentate	**6.2**	**14.15%**
8% Concentration with "tight", RO type membranes:-		
Permeate	**0.9**	**8.00%**
Retentate	**6.3**	**14.70%**

These results are typical of all our observations over several years.

If these parameters were applied to the process as set out by Nielsen, the "wine treatment tank" in the diagram would correspond to Nielsen element 29; the "pressure pump" to element 36; the "membrane separation" to element 35; the "permeate to storage or waste" to element 38. It is clear that the permeate leaving the system would be lower in alcohol and the wine product exiting the process at point 44 in the various figures would be higher in alcohol concentration than the original wine, unless water is added to vessel 29. While this will lower the final alcohol in wine, it is a prohibited process in most parts of the world. In any case, the by-product exiting at point 38 would be lower in alcohol than the original wine.

I have never seen, nor am I aware of any commercial application of the Nielsen process as described in the patent specification.

10. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true. I understand that willful false statements and the like if made herein would be punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code, and may jeopardize the validity of the application or any patent issuing there from.

David Wollan

Date: 24 January 2011

David Wollan

Attachments:

Michaels et al., U.S. Patent 5,817,359

2010 Intervitis Interfructa International Congress in Stuttgart, Germany, paper by Dr Ferrarini (presented by E Bocca)

2010 Intervitis Interfructa International Congress in Stuttgart, Germany, papers by David Wollan, Memstar Pty Ltd.

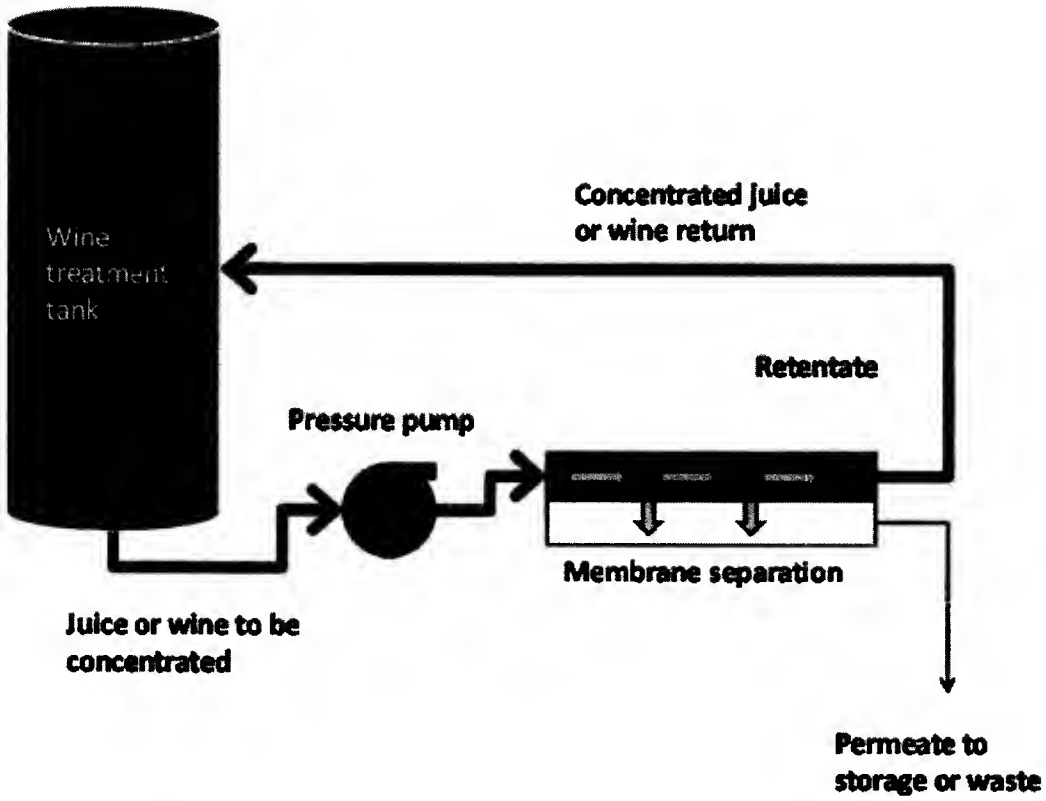
wine feed. It is the inverse of "rejection". In all cases, the alcohol concentration in the permeate is lower than in the wine. In other words, water passes through the membranes more readily than alcohol. In no case was there less than 50% alcohol passage (>50% rejection).

The Vinocon RO-3 and RO-5 as well as the XLE would be considered reverse osmosis elements (Nielsen element 35), with Vinocon RO-3 having the best water/alcohol separation characteristics. The VinoPro and NF90 are typical nanofiltration membranes (Nielsen element 24). It should be noted that in all instances, the alcohol concentration in the permeate is lower than in the feed and, as a corollary, in the retentate. This is clearly contrary to Nielsen's claims of what happens in the Nielsen process.

Membrane type	Pressure kPa	Temp °C	Sample#		Alcohol %v/v	Alc passage	Alc flux ($l/m^2/hr$)	Total Flux ($l/m^2/hr$)
			1	Wine	13.85%			
VinoCon RO-3	6560	19	2	Permeate	6.94%	0.501	0.39	5.68
VinoCon RO-3	4500	20	3	Permeate	8.00%	0.578	0.39	4.86
VinoCon RO-3	2530	21	4	Permeate	9.04%	0.653	0.15	1.62
VinoPro	3080	21	5	Permeate	12.38%	0.894	3.41	27.57
VinoPro	2020	21	6	Permeate	12.98%	0.937	2.10	16.22
VinoPro	1005	22	7	Permeate	13.68%	0.988	0.78	5.68
NF90	3520	22	9	Permeate	9.50%	0.686	1.05	11.05
NF90	2490	22	10	Permeate	10.71%	0.773	0.85	7.89
NF90	1500	22	11	Permeate	12.06%	0.871	0.38	3.16
XLE	6100	19	12	Permeate	6.40%	0.462	0.76	11.84
XLE	4000	19	13	Permeate	8.49%	0.613	0.54	6.32
XLE	2020	19	14	Permeate	11.14%	0.804	0.18	1.58
VinoCon RO-5	6650	20	15	Permeate	7.60%	0.549	1.48	19.46
VinoCon RO-5	4020	21	16	Permeate	7.62%	0.550	0.80	10.54
VinoCon RO-5	1980	21	17	Permeate	11.79%	0.851	0.48	4.05

On another occasion, I tested the alcohol and acid passage performance, comparing the wine concentration effects of two different membrane types.

In these trials a volume of wine was concentrated as shown below such that 8% of the volume was separated as permeate.



That permeate, and the remaining retentate or concentrate (comprising 92% of the original volume), were analyzed for a couple of key parameters – titratable acidity and alcohol. This was repeated for NF and RO type membranes and the results illustrate the different permeabilities of these with regards to wine acids and alcohol.

	Titratable Acidity (g/l as tartaric acid)	Alcohol (% v/v)
Control wine (feed)	5.9	14.10%
8% Concentration with "loose", NF type membranes:-		
Permeate	2.4	13.35%
Retentate	6.2	14.15%
8% Concentration with "tight", RO type membranes:-		
Permeate	0.9	8.00%
Retentate	6.3	14.70%

These results are typical of all our observations over several years.

US005817359A

United States Patent [19]

Michaels et al.

[11] Patent Number: 5,817,359

[45] Date of Patent: Oct. 6, 1998

[54] **METHODS FOR DEALCOHOLIZATION EMPLOYING PERSTRATION**

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[73] Assignee: **Palassa Pty. Ltd.**, Victoria, Australia

[21] Appl. No.: **729,181**

[22] Filed: **Oct. 11, 1996**

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 204,409, Aug. 1, 1994, abandoned.

[30] **Foreign Application Priority Data**

Apr. 30, 1992 [AU] Australia PL2190
Jun. 5, 1992 [AU] Australia PL2808

[51] **Int. Cl.**[6] **C12H 3/04**; B01D 11/04; B01D 61/58

[52] **U.S. Cl.** **426/425**; 210/639; 210/641; 210/644; 426/14; 426/15; 426/16; 426/431

[58] **Field of Search** 210/639, 641, 210/644, 805, 806, 500.36, 640, 650, 651; 426/14, 16, 425, 431, 490, 592, 11, 13, 15

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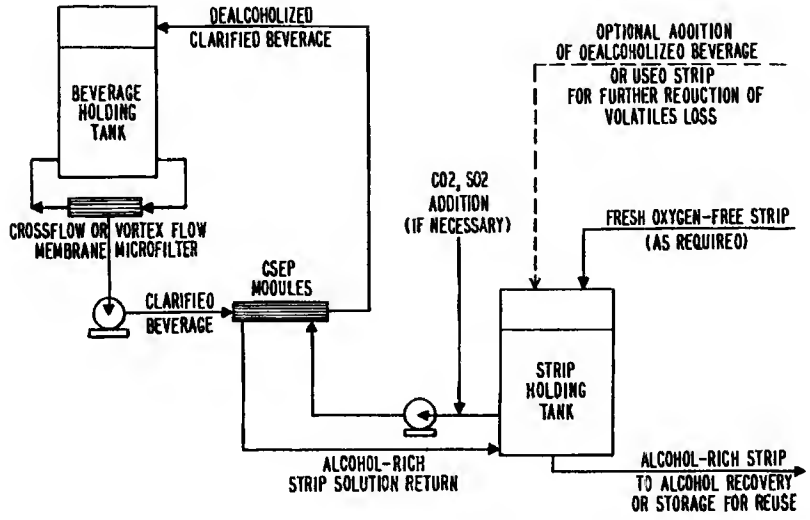
Primary Examiner—Joseph W. Drodge
Attorney, Agent, or Firm—Woodcock Washburn Kurtz Mackiewicz & Norris LLP

[57] **ABSTRACT**

The present invention provides methods and for the dealcoholization of alcohol containing solutions, especially fermented beverages. In preferred embodiments the methods employ a novel controlled-strip evaporative perstraction technique following initial clarification to remove biomass, wherein strip solutions, dealcoholized beverage, or both are recycled for use in the strip solutions for dealcoholization of subsequent batches of feed beverage. In further preferred embodiments additives are introduced into the strip solution to further limit loss of volatile components during perstraction. Biomass removed by the clarification is then returned to the retentate product following perstration.

20 Claims, 1 Drawing Sheet



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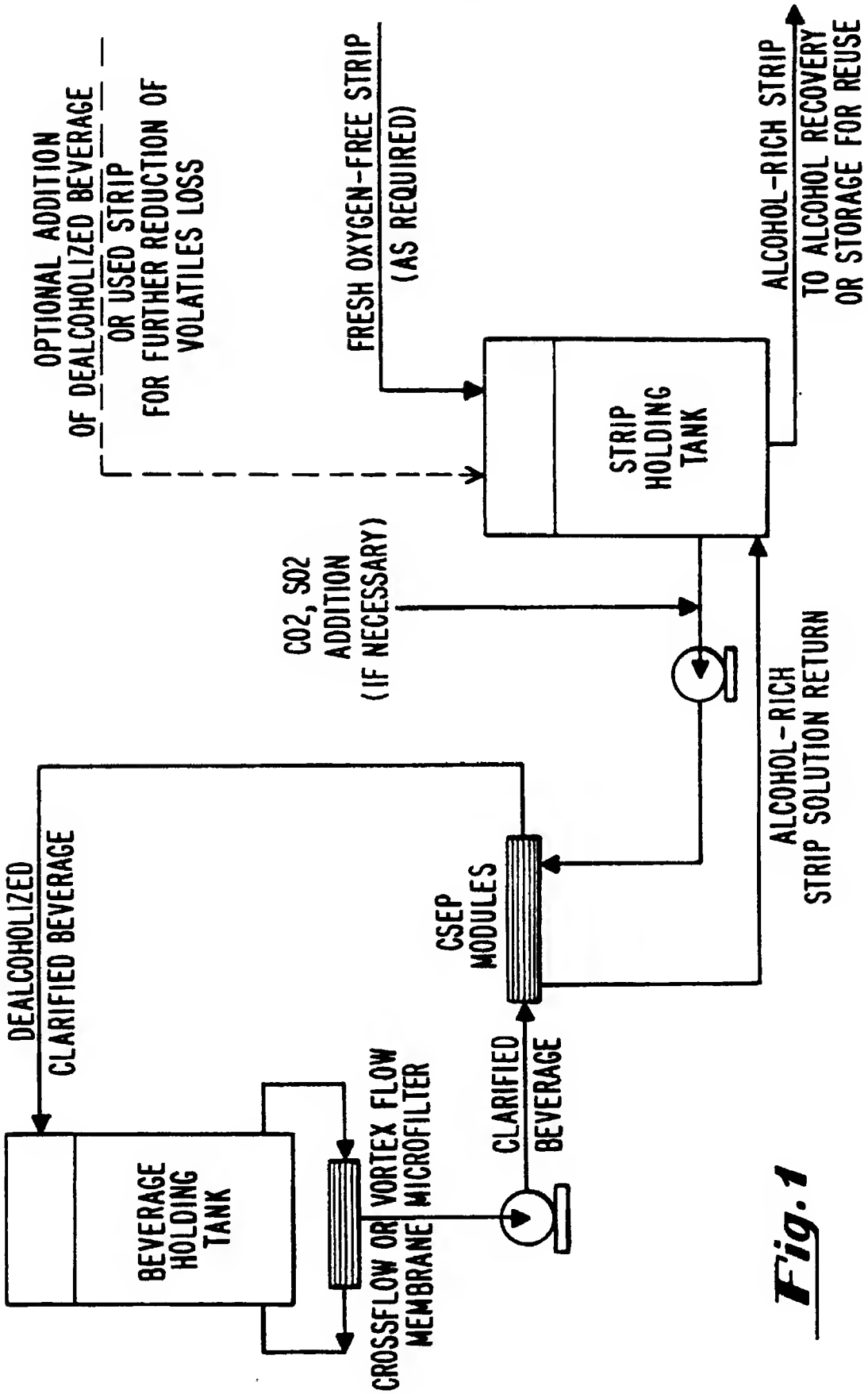
OPTIONAL ADDITION OF DEALCOHOLIZED BEVERAGE OR USED STRIP FOR FURTHER REDUCTION OF VOLATILES LOSS
FRESH OXYGEN-FREE STRIP (AS REQUIRED)
ALCOHOL-RICH STRIP TO ALCOHOL RECOVERY OR STORAGE FOR REUSE
STRIP HOLDING TANK
CO2, SO2 ADDITION (IF NECESSARY)
ALCOHOL-RICH STRIP SOLUTION RETURN
DEALCOHOLIZED CLARIFIED BEVERAGE
CSEP MODULES
BEVERAGE HOLDING TANK
CROSSFLOW OR VORTEX FLOW MEMBRANE MICROFILTER
CLARIFIED BEVERAGE

Fig. 1

METHODS FOR DEALCOHOLIZATION EMPLOYING PERSTRATION

CROSS REFERENCE TO RELATED APPLICATIONS

This application is a continuation-in-part of U.S. application Ser. No. 08/204,409, filed Aug. 1, 1994, now abandoned, which is the National Phase of International Application No. PCT/AU93/00196, filed Apr. 30, 1993, and Australian Application Ser. No. PL2808, filed Jun. 5, 1992, and Australian Application Ser. No. PL2190, filed Apr. 30, 1992.

FIELD OF THE INVENTION

This invention relates to novel methods and apparatus for the dealcoholization of alcohol containing solutions, especially fermented heverages such as beer, ale, hard cider, and the like, without significantly altering the concentrations of other essential components which contribute to the flavor, fragrance, color, shelf-stability, and aging characteristics of such products. In preferred embodiments the methods employ a novel controlled-strip evaporative perstraction technique, wherein strip solutions, dealcoholized beverage, or hoth are recycled for use as a component of the strip solution used for dealcoholization of suhsequent batches of feed heverage. In further preferred emhodiments additives are introduced into the strip solution to further limit loss of volatile components during perstraction.

BACKGROUND OF THE INVENTION

Several processes are currently employed commercially for the dealcoholization of alcoholic beverages, including vacuum-evaporation, memhrane pervaporation, and reverse osmosis. Vacuum-evaporation and memhrane pervaporation suffer from the limitations that they remove not only alcohol, hut also to varying degrees remove other volatile components such as water, flavor-and fragrance compounds, and dissolved gases such as carhon dioxide and sulfur dioxide which are important in controlling the acidity of the beverage, and sterilizing the beverage against attack hy destructive microorganisms. Such composition changes adversely affect the organoleptic properties of the beverage and its appearance and stahility, and require restoration to the dealcoholized product of some or all of the components so removed to attempt to render the product suitable for consumption. Not only is this restoration process difficult and costly, but often it fails to return the product to the level of quality it possessed prior to the dealcoholization process. In addition, such restoration may he prohibited in some countries where it is considered adulteration of a natural product.

Dealcoholization by reverse osmosis removes from the beverage not only volatile low molecular weight components such as water, alcohol, flavor and fragrance components, and dissolved gases such as carhon dioxide and sulfur dioxide, but may also remove certain low-molecular-weight, nonvolatile, nonionic solutes such as organic acids and simple sugars. As a consequence, removal of a significant amount of alcohol from a beverage by reverse osmosis may he accompanied hy the removal of proportionately large amounts of these other membrane-permeable components. Current practice involves dilution of the reverse-osmosis concentrate or retentate with pure water to replace the water removed in the permeate. As a result, the final dealcoholized product is substantially depleted in other memhrane-permeahle solutes, with resulting undesirable

changes in flavor, fragrance, color, acidity, stability, and the like. Restoration of these depleted components by their re-addition to the concentrate in proper proportions to produce a high-quality product is generally difficult, costly, or impossihle.

More recently, another memhrane process, usually described as "osmotic distillation", has been proposed for dealcoholization of such heverages. In this process, the heverage is passed over one surface of a microporous, non-liquid-wettable memhrane, while another liquid in which alcohol is highly soluble (usually termed the "strip solution") is passed over the opposite surface of the membrane. The liquid most frequently proposed for use as the strip solution is pure water. Any volatile components present in the beverage (as well as those present in the strip solution) are free to pass through the pores of the membrane as vapor, and then to condense in the opposing liquid if there is a favorable concentration difference (and thus vapor pressure difference) of that component between the two solutions. Inasmuch, however, as this process effectively constitutes the extraction of a component from one liquid phase into another across a membrane harrier, we refer herein to osmotic distillation as "isothermal membrane perstraction".

An important advantage of isothermal membrane perstraction over evaporation, pervaporation, and reverse osmosis is that, since the vapor pressure of water over most alcoholic ferments is very nearly that over pure water, there is very little transfer of water from the strip solution to the heverage while rapid transfer of alcohol from the beverage to the strip solution is taking place. Hence, the process, in this mode of operation, is highly selective for removal of alcohol relative to water. As a result, the volume of the solution or beverage and the relative proportions of water and all nonvolatile solutes remain virtually the same in the final product as in the original solution or heverage. This process is described in U.S. Pat. No. 5,382,362 to Bowser et al. (and in U.S. application Ser. No. 08/204,409 to A. S. Michaels).

However, isothermal memhrane perstraction has serious limitations which can have adversely affect the properties, characteristics, and quality of a dealcoholized heverage ohtained therehy. Since the membrane employed for that process is freely permeahle to any volatile solute which may be present in either liquid phase in contact with it, the volatile components present in the original beverage are free to leave the beverage and dissolve in the strip solution if the activity of any such solute is higher in the beverage than in the strip solution. Thus, volatile flavor and fragrance components, as well as dissolved gases such as carbon dioxide and sulfur dioxide, which may be present in the original beverage, can be depleted during the perstraction process.

Moreover, any volatile solute which is present initially in the strip solution, and is absent from the initial heverage, may similarly transfer into the beverage, thereby contaminating it. If, as proposed in the Bowser patent, pure water is used as the strip liquid, it will almost invariably contain dissolved oxygen with which it is in substantial equilibrium with the ambient atmosphere. Most ingestihle ferments (wine, beer, and the like) are rendered alcoholic by fermentation of sugars via anaerohic organisms such as yeast, and thus are virtually free of dissolved oxygen. This is an important feature of the fermentation process, since many of the components which are generated in the process are reduced organic compounds which are responsihle for the characteristic fragrance, flavor, color, and acidity of the product. Exposure of these components to elemental oxygen

may, and usually does, result in their oxidation to new chemical entities which differ markedly in organoleptic properties and appearance from those of the original ferment, and frequently have objectionable taste, odor, and/or color. Consequently, exposure of the original beverage during evaporative perstraction to a strip solution comprising pure water containing dissolved oxygen can result in permanent damage to the dealcoholized product, and render it unsuitable for consumption.

Improved methods for the dealcoholization are needed which do not suffer from the aforementioned limitations. This invention is directed to this important end.

SUMMARY OF THE INVENTION

The present invention provides novel methods and apparatus for dealcoholization of alcohol containing solutions, particularly beverages such as wine, beer, ale, hard cider, and the like.

In certain embodiments, the present invention provides methods for the dealcoholization of alcohol containing solutions comprising:

(a) contacting a first side of a hydrophobic microporous membrane with an alcohol-containing feed solution;

(b) contacting the opposite side of said membrane with a strip solution to extract alcohol from said alcohol-containing solution into said strip solution and thereby to provide
an alcoholized strip solution and
a dealcoholized product;

(c) recovering the dealcoholized product and the alcoholized strip solution; and

(d) repeating steps (a) and (b) while recycling at least some of (1) the recovered dealcoholized product or (2) the recovered alcoholized strip solution to the alcohol containing solution of step (a) and/or to the strip solution of step (b).

In some preferred embodiments at least some of the alcoholized strip solution recovered in step (c) is recycled and mixed with the strip solution of step (b), and in other preferred embodiments at least some of the dealcoholized product produced in step (c) is used in the strip solution of step (b). In further preferred embodiments at least some of the alcoholized strip solution recovered in step (c) and at least some of the dealcoholized solution recovered in step (c) is recycled to the strip solution of step (b). In some preferred embodiments the recovered dealcoholized solution is recycled to the alcohol-containing solution of step (a).

Preferred embodiments further comprise selecting a desired amount of alcohol to be removed from the alcohol-containing solution, and using the minimum amount of alcoholized strip solution, dealcoholized solution, or both in the second strip solution to remove the desired amount of alcohol.

Preferably, the strip solution is pure water, which has preferably been deoxygenated by boiling and flushing with a gas, or by addition of reducing agent.

In further preferred embodiments the strip solutions are saturated with carbon dioxide, preferably by bubbling in carbon dioxide gas, or by addition of solid carbon dioxide.

In yet further preferred embodiments a stabilizing agent, preferably sulfur dioxide, sodium bisulfite, potassium bisulfite, sodium metabisulfite, or potassium metabisulfite is added to the strip solutions.

In preferred embodiments the alcohol-containing feed solution is a beverage, particularly wine, beer, ale, or hard cider. Preferably, the alcohol-containing feed solutions are clarified prior to contacting the hydrophobic microporous membrane, preferably by crossflow membrane microfiltration or vortex flow membrane microfiltration.

The hydrophobic microporous membrane is preferably comprised of polyethylene, polypropylene, polyalkylsilane, polyalkarylsilane, polysulfone, or polycarbonate, with polypropylene being particularly preferred.

Also provided in accordance with the present invention are apparatus for performing the controlled-strip evaporative perstraction methods.

BRIEF DESCRIPTION OF THE DRAWINGS

The numerous objects and advantages of the present invention may be better understood by those skilled in the art by reference to the accompanying FIG. 1, which is a basic illustration of a preferred embodiment of the dealcoholization method of the invention.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

This invention provides novel methods and apparatus for reducing the alcohol content of alcohol-containing solutions. The "controlled-strip evaporative perstraction" (CSEP) methods of the invention selectively remove alcohol from alcohol-containing solutions, such as alcoholic beverages, while avoiding the serious limitations of existing osmotic distillation methods. Specifically, the methods of the invention remove alcohol without concurrently removing significant quantities of other essential components, and without subjecting the product to destructive oxidative deterioration by exposure to atmospheric oxygen.

In preferred embodiments, the solution from which alcohol is to be removed is a beverage, especially a wine, beer, ale, or the like. However, the methods of the invention are amenable to the partial or complete dealcoholization of a wide variety of alcohol containing solutions.

In accordance with the multistep strip-recycling dealcoholization methods of the present invention, the absolute and relative concentrations of all non-alcohol volatile components in the strip solution are advantageously maintained as close as possible to those initially present in the alcohol-containing feed solution. Thus, the methods of the present invention minimize the extraction of desirable volatile components from the feed during the dealcoholization process. These desirable volatile components include the volatile flavor and fragrance components which impart the desired organoleptic properties to the alcoholic beverage; volatile acidic components, such as carbon dioxide, which impart the proper acidity to the product; and volatile stabilizing additives, such as sulfur dioxide, which insure product sterility and protect against oxidative degradation of the product on storage and aging.

The methods of the present invention advantageously maximize the difference in alcohol activity and/or alcohol concentration between the feed solution or the dealcoholized product and the alcohol activity and/or alcohol concentration in the strip solution on the opposite side of the membrane. Accordingly, the methods of the invention maximize the rate at which alcohol can be selectively extracted from the feed and transferred to the strip solution.

In preferred embodiments of the methods of the invention, pure water is used as the initial strip solution. It is preferable that the strip solution is devoid of any foreign volatile component, such as dissolved oxygen, which can transfer into the feed and/or product and damage the product. It is also preferable that the strip solution be unable to

extract from the feed solution appreciable amounts of any critical volatile component of the feed solution, such as carbon dioxide and/or sulfur dioxide, the removal of which might cause irreversible damage to the product.

Accordingly, in preferred embodiments of the methods of the invention, the water to be used as the initial strip solution is pretreated to remove dissolved oxygen, and to dissolve therein sufficient carbon dioxide, and sulfites (i.e., sulfur dioxide) to provide concentrations or activities of these solutes substantially equal to those present in the alcohol-containing feed solution.

In some preferred embodiments, a first side of a hydrophobic microporous membrane is contacted with an alcohol-containing feed solution, and the opposite side of the membrane is contacted with a strip solution, and a desired amount alcohol is perstracted from the feed solution into the strip solution. As used herein, the term "contacting" means directly or indirectly causing the moieties to be contacted to come into physical association with each other.

Typically, the feed solution is circulated from a holding tank through a membrane unit and is returned to the holding tank for further recirculation through the membrane until the desired alcohol content is achieved. However, in some instances it is preferable to subject the feed beverage to only a single pass through the membrane, to avoid spoilage of the beverage due to the turbulence of the recirculation process. In such instances, the feed solution is not returned to the holding tank, but rather is collected and stored.

The membrane unit can be any of a variety of devices known in the art to be useful in osmotic distillation processes. In preferred embodiments the membrane unit is a container which provides for the through-circulation of strip and feed solutions on opposite sides of the microporous membrane. One suitable membrane unit is the "Liquicel" unit, manufactured by Hoechst Celanese, Charlotte, N.C.

The microporous membrane can be any of the variety of hydrophobic, microporous membrane materials known to be useful in osmotic distillation. Generally, such membranes are derived from polymeric materials which do not include hydrophilic groups in their network. Examples of these membranes include polyolefins such as polyethylenes, polypropylenes, polyisopropylenes, polyvinylidenes, or mixtures thereof.

Suitable hydrophobic membranes include microporous polypropylene membranes in the form of flat sheets or hollow fibers manufactured by Hoechst Celanese Separations Products Division, Charlotte, N.C., and sold under the name "Celgard", particularly "Celgard 2500", and those manufactured by AKZO NV, Wuppertal, Germany, and sold under the name "Accurel." It is preferable that the membrane be in the form of a hollow fiber, as it has been found that hollow fiber membranes have superior fluid management properties, and are more efficient due to their greater surface area relative to flat sheet fibers.

The initial strip solution is preferably pure water which has been deoxygenated. As used herein, the term "deoxygenated" means containing substantially no molecular oxygen. Deoxygenation of the strip solution can be achieved by any of the several methods known in the art, including, for example, simple steam-stripping of the water by allowing it to boil (either at atmospheric pressure, or under reduced pressure), flushing the vapor space with an inert gas such as nitrogen or carbon dioxide, and storing the strip solution in a vessel blanketed with such gas.

Another suitable deoxygenation technique is to subject the strip solution to pervaporative stripping under reduced pressure using a microporous membrane device, such as a microporous polyolefin hollow fiber module such as those sold by Hoechst Celanese Separations Systems Division. Another suitable method is to add to the strip solution a reducing agent which will combine with oxygen present to form a chemically inert, nontoxic, nonvolatile oxidation product. Preferred reducing agents for this purpose are alkali metal sulfites such as sodium bisulfite, sodium metabisulfite, potassium bisulfite, or potassium metabisulfite, since these additives have been approved for use as stabilizers in wines and other alcoholic beverages.

In preferred embodiments the strip solution is saturated with carbon dioxide. Since the initial beverage to be dealcoholized will usually be saturated with carbon dioxide, similar saturation of the strip solution will assure that there will be no transfer of carbon dioxide into or out of the beverage, and thus no change in beverage acidity. Saturation of the strip solution with carbon dioxide can be accomplished by any of several known techniques. One suitable technique is by bubbling carbon dioxide gas into the liquid. Another suitable technique is by adding solid carbon dioxide ("dry ice") to the liquid.

For feed solutions which contain sulfites, it is preferable that there be no net transfer of sulfur dioxide into or out of the feed solution during dealcoholization. In preferred embodiments this is achieved by maintaining the sulfite content of the strip solution is as close as possible to the sulfite content of the feed solution from which alcohol is to be removed. This can be accomplished by analyzing the alcohol-containing feed solution to ascertain its sulfite content, and adding an appropriate amount of sulfur dioxide or metabisulfite to the strip solution to match that of the feed solution. The sulfur dioxide or metabisulfite can be added to the feed solution before or after saturation with carbon dioxide, it only being necessary that the feed solution contain the proper sulfite and carbon dioxide content before dealcoholization across the microporous membrane.

In typical osmotic distillation processes, volatile flavor and fragrance components present in the feed solution are free to transfer by evaporation and condensation from the feed solution into the strip solution. One way of minimizing the loss of these desirable components is to analyze the feed solution to ascertain the identities and relative proportions of these components, and to then add the proper amounts of the components to the strip solution. This is a costly and arduous analytical task. In preferred embodiments of the methods of the present invention, however, the strip solution is composed of pure water to which has been added either strip solution or dealcoholized product recycled from the perstraction of a previous batch of the identical feed solution. Accordingly, the methods of the present invention provide a strip solution containing the desired volatile flavor and fragrance components in the proper proportion, eliminating the need to ascertain either the identities or the proportions of these desired components.

In preferred embodiments of the present invention the loss of these volatile flavor and fragrance components from the feed solution is further limited by operating the perstractive method in a "batch-recycle mode". In this mode a fixed volume of feed solution is recycled through the membrane unit, while simultaneously recycling a fixed volume of strip solution. The amount (i.e., volume) of strip solution that is so recycled is selected to be the minimum required to allow removal of the desired fraction of alcohol from the product, thus minimizing loss of volatile components. For example, for many high-quality varietal wines, the objective of dealcoholization is to reduce the alcohol concentrations of

excessively-high-alcohol-content ferments (so called "hot" wines; typically about 15% alcohol by volume) to slightly lower levels where they are more palatable (typically about 12% by volume). This can be accomplished using a batch recycle mode wherein a first batch of feed wine is perstracted using a strip solution that is alcohol-free at the beginning of the run, and which contains 10% alcohol by volume at the end of the run. This will typically require about 30 liters of strip solution for each 100 liters of feed wine. The maximum reduction in the concentration of flavor and fragrance volatile components in the final product will thus be only about 30%. The 10% alcohol strip solution remaining after the first batch has been treated is then used as a first strip solution for a second batch of feed wine. This is then followed by replacement of the alcohol-rich strip with fresh, alcohol-free solution thus further reducing the loss of flavor and fragrance volatile components. Such a "sequentially strip-replacement" strategy can keep the loss of volatile components from the product at negligibly low levels. Thus, in one aspect the invention provides perstraction methods wherein at least some of the alcoholized strip solution produced from the dealcoholization of an initial batch of feed solution is used in the strip solution for dealcoholization of succeeding batches of feed solution.

In another aspect of the invention a further beneficial reduction in volatile components loss during dealcoholization can be achieved by retaining a fraction of the dealcoholized product generated from a prior batch of the same feed stock, and adding this solution to the primary strip solution employed during the first stage of dealcoholization of the subsequent batch. In this manner, the concentration of all volatile components (except alcohol) in the strip solution is elevated, and thus the rate of transfer of such volatile components from the feed to the strip is thereby reduced.

Further aspects of the present invention are directed toward the dealcoholization of superior-quality vintage wines, which require fermentative aging under special conditions to develop the desired flavor, fragrance mouth-feel and related attributes so important to their marketability. Such wines, after primary vat-fermentation, are separated from the lees (fruit residue and fermentation biomass), but are allowed to retain in suspension low concentrations of non-viable yeast cells. These cells contain a variety of active enzymes which can act upon residual sugars, polysaccharides, organic acids, and flavor and fragrance precursors present in the ferment and catalyze their transformation into new compounds which are essential to the development of superior flavor and "nose" in the final aged product. This process requires the presence of this biomass, and is accomplished by barrel-aging of the vat-ferment. For optimal conduct of this secondary fermentation, it is important that the suspended cells remain intact, and that the alcohol content of the ferment be maintained within a narrow concentration range. Vintages which yield fruit of high sugar content tend to produce vat ferments containing excessively high alcohol concentrations, so the reduction of the alcohol content of such ferments (so-called "trimming") is necessary to achieve optimal in-barrel secondary fermentation.

Most conventional dealcoholization processes are quite destructive of such suspended cells, and thus retard or prevent this secondary fermentation process. Concentration by evaporative membrane perstraction would mitigate this cell damage, were it not for the problem that suspended particulates tend to foul the membranes and make dealcoholization difficult or impractical. This problem can be avoided by subjecting the primary ferment to membrane prefiltration in order to temporarily remove the suspended biomass without significant cell damage, followed by controlled dealcoholization of the clarified ferment, and then returning the biomass to the dealcoholized product. The resulting product now contains the optimal alcohol concentration, and the fully active biomass content, required for optimal in-barrel secondary fermentation.

Prefiltration is carried out using either microfiltration or ultrafiltration membranes, and modular devices containing such membranes which provide tangential flow or vortex flow in the feed channels, permitting high-flow separation of the biomass from the ferment at low pressure, with minimal damage to the suspended cells. Removal of as much as 90% of the liquid volume as clarified filtrate can be achieved in this manner, yielding a concentrated biomass suspension which can readily be blended back with the dealcoholized filtrate. Since all other volatile components present in the primary ferment are conserved in this process, the product provided for barrel fermentation is virtually undistinguishable from the initial ferment except for its reduced alcohol content. Via this novel hybrid process, the production of superior barrel-aged varietal wines is now possible.

The invention is further illustrated by way of the following examples which are intended to elucidate the invention. These examples are not intended, nor are they to be construed, as limiting the scope of the disclosure.

EXAMPLES

Illustration of Controlled-Strip Evaporative Perstraction in Batch Recycle Mode

FIG. 1 shows a flow-diagram illustrating a preferred embodiment of the present invention. The dealcoholization apparatus consists of a feed holding tank containing an alcohol-containing solution, typically a beverage; a crossflow or vortex flow microfilter loop for clarifying the alcohol-containing solution; one or more controlled-strip evaporative perstraction (CSEP) modules, each of which include one or more hydrophobic microporous membranes; a pump and attendant piping for pumping the alcohol-containing feed solution through the CSEP modules and returning at least a portion of the alcohol-depleted solution to the feed beverage holding tank; a strip holding tank containing a strip solution; a pump and attendant piping for pumping the strip solution through the membrane module and returning at least a portion of the alcoholized strip solution to the strip holding tank; an inlet in the strip holding tank for adding fresh strip solution to the strip holding tank; an inlet in the strip holding tank for adding alcoholized strip solution or alcohol-depleted solution to the strip holding tank; an inlet for adding additives such as carbon dioxide or sulfur dioxide to the strip solution, located either in the strip holding tank or in the connecting line between the strip holding tank and the CSEP modules; and an outlet for removing alcoholized strip solution from the strip tank.

Initially, a first batch of alcohol-containing feed beverage is placed in the beverage holding tank, and a fresh, deoxygenated, alcohol-free water strip solution is placed in the strip holding tank. The strip solution is saturated with carbon dioxide, and, if necessary, sulfur dioxide is added to the strip solution to match the sulfite content of the feed beverage. This can be done either in the strip holding tank, or in the line from the strip holding tank to the CSEP modules.

The feed beverage is pumped from the beverage holding tank into a crossflow or vortex flow microfilter loop, wherein it is passed through a crossflow or vortex flow microfilter, which clarifies the feed beverage by filtering out large

suspended particles, which are typically cells or other biomass present in the beverage. The filtered biomass is returned to the beverage holding tank, and the clarified feed beverage is pumped through the controlled-strip evaporative perstraction (CSEP) modules, where alcohol is perstracted from the feed beverage to the strip solution, which is on the opposite side of the membrane. The dealcoholized clarified beverage is then typically returned to the beverage holding tank for another circulation through the system, or the pumping of feed beverage is ceased after a single pass through the perstraction unit. The strip solution is simultaneously pumped from the strip holding tank, through the CSEP modules, and is returned to the strip holding tank. At the end of the initial run, a second batch of fresh feed beverage is placed in the beverage holding tank, and a second strip solution is placed in the strip holding tank. The second strip solution is composed of fresh, deoxygenated strip solution, and at least some of the dealcoholized beverage from the initial run, or some of the alcoholized strip solution produced in the initial run, or both. The presence of these components in the second strip solution reduces the loss of volatile components to minimal values. The amount volume of strip solution that is recycled is selected to be the minimum required to allow removal of the desired fraction of alcohol from the product. The second feed beverage is then dealcoholized using the second strip solution, followed by dealcoholization with fresh strip solution. The process of retaining used strip solution and/or dealcoholized beverage for use in the dealcoholization of succeeding batches of beverage is repeated until the desired amount of dealcoholized beverage is produced.

It will be appreciated that the benefits of the foregoing "batch mode" embodiment can also be obtained by using a continuous process, wherein fresh feed and fresh strip solution are continuously added to the system, and appropriate amounts of dealcoholized beverage and used strip solution are continuously added to the strip solution, and product continuously removed.

Each of the patents, publications, and other published documents mentioned or referred to in this specification is herein incorporated by reference in its entirety.

As those skilled in the art will appreciate, numerous changes and modifications may be made to the preferred embodiments of the invention without departing from the spirit of the invention and the scope of the appended claims.

What is claimed is:

1. A method of reducing the alcohol content of an alcohol-containing solution comprising:

(a) clarifying a fermented alcohol-containing feed solution to remove suspended biomass;

(b) contacting a first side of a hydrophobic microporous membrane with the alcohol-containing feed solution;

(c) contacting the opposite side of the membrane with a strip solution to extract alcohol from said alcohol-containing solution into said strip solution and thereby to provide
an alcoholized strip solution and
a dealcoholized product;

(d) recovering the dealcoholized product and the alcoholized strip solution;

(e) repeating steps (b) and (c) while recycling at least some of (1) the recovered dealcoholized product or (2) the recovered alcoholized strip solution to the alcohol containing solution of step (a); to the strip solution of step (b); or to both the alcohol containing solution of step (a) and to the strip solution of step (b); and

(f) returning at least some removed biomass to the dealcoholized product.

2. The method of claim 1 wherein at least some of the dealcoholized product recovered in step (d) is recycled to the strip solution of step (c).

3. The method of claim 1 wherein at least some of the alcoholized strip solution recovered in step (d) is recycled to the strip solution of step (c).

4. The method of claim 1 wherein at least some of the dealcoholized product recovered in step (d) and at least some of the alcoholized strip solution recovered in step (d) is recycled to the strip solution of step (c).

5. Then method of claim 1 wherein at least some of the dealcoholized product recovered in step (d) is recycled to the alcohol-containing feed solution of step (b).

6. The method of claim 1 further comprising the step of selecting a desired amount of alcohol to be removed from the alcohol-containing feed solution by steps (b), (c) and (e).

7. The method of claim 6 wherein the amount of alcoholized strip solution, dealcoholized product, or both recycled to the strip solution of step (c) is the minimum amount required to remove the desired amount of alcohol.

8. The method of claim 1 wherein the strip solution is pure water.

9. The method of claim 1 further comprising deoxygenating the strip solution prior to contacting said strip solution with said membrane.

10. The method of claim 9 wherein the strip solution is deoxygenated by boiling and flushing with a gas, or by addition of reducing agent.

11. The method of claim 1 further comprising saturating the strip solution with carbon dioxide.

12. The method of claim 11 wherein the strip solution is saturated with carbon dioxide by bubbling in carbon dioxide gas or by addition of solid carbon dioxide.

13. The method of claim 1 further comprising adding a stabilizing agent to the strip solution.

14. The method of claim 13 wherein the stabilizing agent is sulfur dioxide, sodium bisulfite, potassium bisulfite, sodium metabisulfite, or potassium metabisulfite.

15. The method of claim 1 wherein the alcohol-containing solution is clarified by crossflow membrane microfiltration or vortex flow membrane microfiltration.

16. The method of claim 1 wherein the alcohol-containing solution is a beverage.

17. The method of claim 16 wherein the beverage is wine, beer, ale, or hard cider.

18. The method of claim 1 wherein the hydrophobic microporous membrane is selected from the group consisting of polyethylene, polypropylene, polyalkylsilane, polyalkarylsilane, polysulfone, and polycarbonate membranes.

19. The method of claim 18 wherein the hydrophobic microporous membrane is a polypropylene membrane.

20. An improved osmotic distillation method for reducing the alcohol content of an alcohol-containing solution comprising clarifying a fermented alcohol-containing feed solution; deoxygenating a strip solution, saturating the strip solution with carbon dioxide, dealcoholizing the alcohol-containing solution and simultaneously alcoholizing the strip solution; and recycling at least a portion of the alcoholized strip solution or the dealcoholized feed solution for use in the strip solution.

* * * * *

INTERNATIONAL
IVIF-CONGRESS 2010

24.–28.03.2010 NEW STUTTGART TRADE FAIR CENTRE

CONGRESS-PROGRAMME 2010

INTERVITIS INTERFRUCTA

Quality · Sustainability · Marketing: Impact on Innovation

Ladies and Gentlemen,

from March, 24th to 28th the INTERVITIS INTERFRUCTA and the IVIF-Congress will take place in the most attractive and most modern congress centre of Europe, the New Stuttgart Trade Fair Centre, next to the Stuttgart airport which is comfortably accessible for both, international and domestic visitors.

The IVIF-Congress events are organized under the patronage of the OIV, the International Organisation for Vine and Wine in Paris, and stand under the motto "Quality · Sustainablitiy · Marketing: Impact on Innovation".

The programme of the IVIF-Congress addresses practising experts, consultants, scientists and representatives of companies from the wine, fruit and spirits sector. The manifold congress activities for the wine sector will be untied under the 60th Grape and Wine Congress.

This year, the congress has been newly structured: On four days, international specialized lectures will be sectioned in compact modules taking place daily from 9:00 to 12:00 a.m. and 3:00 to 6:00 p.m. This offers the possibility to combine the attendance of the congress with a visit of the fair and flexibly organize one's personal programme.

Tickets for the attendance of the congress activities can be comfortably booked in the online shop of the German Winegrowers' Association under http://shop.dwv-online.de. Against payment of the attendance fee you will receive an entrance code for the visit of the INTERVITIS INTERFRUCTA 2010 free of charge. We are pleased to answer all your questions. Please contact us via e-mail under ivif@dwv-online.de

On behalf of the German Winegrowers' Association, the New Stuttgart Trade Fair Centre and all co-organizers of the IVIF-Congress we cordially invite you to visit the INTERVITIS INTERFRUCTA and attend the events of the IVIF-Congress. The programme will certainly include events which will meet your personal interests. We are looking forward personally welcoming you in Stuttgart!

Your

Dr. Rudolf Nickenig
Overall direction IVIF-Congress

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INTERVITIS INTERFRUCTA

Wednesday, March 24th, 2010

GLOBAL CHALLENGE FOR THE WINE AND FRUIT SECTOR

Moderation: Dr. Rudolf Nickenig

10:00 a.m. Address of Welcome by the Organiser
Dr. Rudolf Nickenig, Secretary General, German Winegrowers' Association, Germany

10:05 a.m. Sustainability Strategy of Baden Wurttemberg
Representative of the government of Baden Wurttemberg, Germany

10:15 a.m. Germany's Most Green Exhibition Centre – Stuttgart as an Example for Sustainability
Ulrich Kromer von Baerle, Landesmesse Stuttgart GmbH, Germany

10:25 a.m. OIV Guidelines for Sustainable Vitiviniculture: Production, Processing and Packaging of Products – Environmental Issues
Yves Bénard, President of the International Organisation of Wine and Vine (OIV), France

10:55 a.m. Viticulture and Climate Change: Global Challenge – Concrete Actions
Prof. Dr. Klaus Töpfer Founding Director, Institute for Advanced Sustainability Studies (IASS) Potsdam, Germany

11:25 a.m. Environmental Sustainability in Miguel Torres, S.A.
Mireia Torres, Miguel Torres Winery, Spain

11:50 a.m. German Wine – Preserve the Future
Norbert Weber, President, German Winegrowers' Association, Germany

12:20 a.m. Disscussion

12:30 a.m. End

ALCOHOL MANAGEMENT

Moderation: Prof. Monika Christmann

3:00 p.m. Introduction

3:05 p.m. Managing Wine Flavor Development and Alcohol Content in the Vineyard: A California Perspective
Dr. Nick Dokoozlian, Vice president – Viticulture, Chemistry and Enology, E&J Gallo Winery, USA

3:30 p.m. The Lighter Side of Alcohol: Taking Control of the Highs and Lows of Alcohol in Wine
Prof. Sakkie Pretorius, Australian Wine Research Institute (AWRI), Australia

3:55 p.m. Alcohol Management in the Winery
David Wollan, Memstar Pty Ltd, Australia

4:20 p.m. Coffee Break

4:40 p.m. The Role of Ethanol and Glucose on the Headspace Partitioning of Volatile Compounds
Anthony L. Robinson, Separation Science Laboratory, Murdoch University, Australia

5:05 p.m. Consumer Reaction towards Partial Dealcoholization
Dr. Sophie Meillon, France

5:30 p.m. Low Alcohol Quality Wines: What Perspectives on the French Market?
Prof. François d'Hauteville, UMR MOISA, France

5:55 p.m. End

5:25 p.m. **A Microbiological Systems Comparison between Integrated, Organic and Biodynamic Plant Treatment Strategies**
Dr. Christian von Wallbrunn, Forschungsanstalt Geisenheim, Germany

5:50 p.m. **Organic Wine Production: Implementing Oenological Rules on EU-Level**
Dr. Uwe Hofmann, ECO-CONSULT, Germany

6:00 p.m. **End**

PROCESS TECHNOLOGY FOR PROCESS CONTROL AND ALCOHOL MANAGEMENT

Moderation: Prof. Dr. Monika Christmann and Dr. Gerd Scholten

2:00 p.m. **Poster Session**

3:00 p.m. **Introduction**

3:05 p.m. **Direct Grape Processing and Juicing in the Vineyard**
Prof. Dr. Tilo Hühn, ZHAW Zentrum für Getränke und Aromaforschung, Switzerland

3:25 p.m. **Carboxymethylcellose for Crystal Stabilisation**
Dr. Gerd Scholten, DLR Mosel, Germany

3:45 p.m. **On-line Measurement of Electrochemical Potential on Must and Wine: Applications and Examples in Wineries**
Mariano Fernandez, DOLMAR, Spain

4:05 p.m. **Coffee Break**

4:20 p.m. **Sensory Impact of Ethanol – the Hidden Sensory Giant**
Prof. Dr. Ulrich Fischer, DLR Rheinpfalz, Germany

4:40 p.m. **New Membrane Approach to Optimise the Concentration of Dissolved Gases in Wine and to Reduce the Alcohol Levels of Wine**
Dr. Oliver Schmidt, LVWO Weinsberg, Germany

5:00 p.m. **Technological Options for Dealing with High Alcohol Wines**
David Wollan, Memstar Pty Ltd, Australia

5:20 p.m. **Many years' Experiences on the Dealcoholization Processes Using Membranes Techniques to Guarantee the Quality and Sustainability of the Wine**
Prof. Roberto Ferrarini, Università degli Studi di Verona – Dipartimento di Scienze, Tecnologie e Mercati, Italy

5:40 p.m. **Tasting**

6:00 p.m. **End**

INTERNATIONAL IVIF-CONGRESS 2010

24.–28.03.2010 NEW STUTTGART TRADE FAIR CENTRE

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Deutscher Weinbauverband e.V., Dr. Rudolf Nickenig

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Prozesstechnik - Prozesskontrolle und Alkoholmanagement

Process Technology for Process Control and Alcohol Management

27.03.2010

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Internationale Technologiemesse für Wein, Obst, Fruchtsaft und Spirituosen
INTERVITIS INTERFRUCTA
Neue Messe Stuttgart
24.–28. März 2010

Many years' experiences on the dealcoholization processes using membranes techniques to guarantee the quality and sustainability of the wine.

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Abstract

In this work are shown the results of several experiments performed with different membrane techniques (contactor, osmotic membranes, nanofiltration) on different white and red Italian wines with different degrees of alcohol removal, reporting chemical analysis of the major and more quality and stability related components of wine (varietal and fermentative aromatic fractions), as well as performances data of the various tested membrane processes.

Introduction

Currently it is increasingly common to find an excessive alcohol content in wine production, due, first, to the climate trends of the last harvests that led to this effect even in the temperate regions. Also the search for advanced states of ripening of grapes, aimed at obtaining structured wines with a strong territorial characterization, can result in an excessive presence of sugars in the musts and, consequently, of alcohol in the wine; such situation frequently affects high-range wines.
The excess of alcohol content, which more and more frequently can exceed the limit of 15%, does not guarantee the qualitative equilibrium, and represents for the consumer the most critical factor in assessing the product's wholesomeness.
To overcome this critical issue, the European Union with the entry into force of the new CMO, has allowed wines' alcohol removal through physical processes for up to 2 alcohol percent. On the contrary in the United States the problem of alcohol excess is resolved by adding water directly to the musts with high sugar content (over 22 ° Brix), thus restoring that water due to dehydration of the berry before harvest. This technique has different qualitative effects compared to the alcohol removal performed on the wine, and it may also be less culturally acceptable by the consumers and the producers of the more traditional wine areas such as the European ones.
Concerning alcohol removal techniques, there are different technological approaches: decreasing must's potential alcohol content (removal of sugar concentration), or separating a fraction of the alcohol from the wine.

In this case the reduction of wines' alcoholic content can be made with different techniques of physical separation: Spinning Cone Column, distilling the RO (reverse osmosis) permeate, pervaporation, use of "Contactor" membranes.
In particular, the "Contactor" membrane technique, by itself or combined with other separating actions, enables the implementation of highly performing alcohol removal processes, some of which have been researched and tested (the relative results are reported below).
Moreover, the aim of this investigation is the control of the volatiles content variation due to these technological test and possibly justify the maintaining of several quality-supporting compounds that, according to the actual literature knowledges, can be important and characteristic of some famous Italian wine types, like a fresh fermented white 'Moscato d'Asti' – both as still and sparkling wine in different experimental approaches -, two three-year old still red 'Sfursat' wines from partially dried Nebbiolo grapes of Valtellina area in Northern Italy close to the Switzerland, a two-year old Chianti of Tuscany and a two-year old Valpolicella of Verona Province.

Materiali e metodi

Dealcoholization system

The dealcoholization technique studied in this work is the "Contactor"; it involves the treatment of wine with hydrophobic membranes that, used between the product and an extractive solution (water), creates a gaseous layer (gaseous membrane) trough which the most volatile compounds pass. Among these compounds, the most representative is alcohol (Fig. 1). The difference between the vapour pressure of alcohol in wine and the extractive solution permit the passage of ethanol, in form of vapour, trough the gaseous membrane and then the dissolution in the water of the extractive circuit.
The membranes used in this work have been supplied by Juclas (Verona – I); they're PTFE (Teflon®) hollow fibers (ext. Ø 1 mm, thickness 200 µm).
The system used for the study of the effects of dealcoholization on wine composition is a pilot plant with a 0,2 m^2 membrane; the industrial plant, used to remove up to 2% of alcohol, has a membrane with the surface of 20 m^2; the flow rate of wine and water in contact with the membrane is of 0,1-0,3 ms^{-1}. All the treatments have been carried out at environmental temperature. The vaporizing efficiency of the membrane is between 0,1 and 0,2 l m^{-2} h^{-1} of anhydrous acohol at 20°C .

Study of the effects of the dealcoholization rate with a contactor membrane on the composition of wines.

The experiences have been made just after the end of fermentation on wine from Moscato Fior d'Arancio grapes, produced in the zone of Colli Euganei, Padua (I).
We performed four dealcoholization experiences at 4 different alcohol removal rates: 1.4 , 2.7 , 5,1 and 7,1 % v/v.
Industrial tests of low dealcoholization (up to 2% removal) on some typical Italian wines.
Considering the recent legal changes with the new OCM, it's allowed a dealcoholization up to 2% v/v.
Here are reported some dealcoholization experiences lowering the acohol degree up to 2° (maximum allowed by the European law) of some typical Italian wines: a red wine

"Valpolicella" (produced in the Verona region), 2 "Sfurzat" from Valtellina (Sondrio region), 1 "Chianti" produced in Tuscany (Siena).

Analytical methods

Analytical determination performed on wines have been made following the European [CEE, 1990] and OIV [OIV,2007] methods.
It has been evaluated tartaric stability by the T°sat and the Mini contact methods using the Criosmall instrument (Steroglass – Pe – I).
Aroma profiles have been investigated after proper enrichment techniques due to the substances and/or relevant concentrations we are going to consider, like SPE (Solid Phase Extraction) using a very exhaustive adsorption cartridge (ENV+), but mostly with headspace-SPME (Solid Phase Microextraction) with a three-phases fiber [see some literature references and some recently optimised methods by UIV Co., Verona]. All the compounds enriched have been quantified by HRGC-MS. In few cases we considered also the quantification of methanol, acetaldehyde and ethyl acetate by direct injection in GC-FID, after a preliminary distillation, 1:4, v/v, of the matrix (capillary column CPWAX-57, 30 m x 0.3 mm x 0.32 µm). Acetaldehyde and ethyl acetate could contribute in fact to some off-flavour as a grassy and vinegar-like note, respectively.
Analytical attention has been payd mainly to the following compounds classes:
- secondary compounds – the very important quality-imprinting contributors -, like fermentation esters (acetates of higher alcohols and other alcohols; different ethyl esters, mostly the fatty acid ones from C4 to C10), the so-called 'leaves' C6-alcohols, some peculiar fermentation scent-active compounds like 2-phenylethanol and methionol, some derivatives from hydroxycinnamic acids as vinyl- and ethylphenols;
- varietal compounds, basically: monoterpenols, monoterpenes, monoterpenyl oxides, monoterpenyl ethylethers (usually in traces, but potent aroma contributors!), norisoprenoids (C13-carotene derived compounds) and some aryl compounds;
- different storage/ageing compounds mainly derived from the barrels and relevant co-operage techniques.

Sensory Analyses

Moscato wines, grown from study of the effects of the dealcoholisation, have been sensory analyses using 27 trained panelists. The descriptors chosen for sensory analysis were: bitter, sweet, salty, acidity, fruity, floral, honey, vegetable, structure, persistence. Data were collected through a "unipolar non-structured" scale (M. Ubigli, 2004) with values from 0 to 100. Results were treated statistically with Friedman test.

Results and discussion

Study of the effects of the deacoholization rate with contactor membrane on the wine composition

The main composition of the wines analyzed for the study of dealcoholization effects are reported in Tab. 1.

Data show, as predictable, an increasing of the fix components of wine which is proportional to the concentration due to the ethanol removal and consequent volume reduction.
In Table 1 are also reported the Tsat test results of the dealcoholized wines; the Tsat decreases proportionally to the dealcoholization rate.
The following sentences regards the aroma compounds: (Tab 2)
A quite linear decrease happens both for the acetates (ethyl acetate enclosed even if with a partially quantittive evaluation) and the ethyl esters of fatty acids reaching about 1/5 content for C4-C8 acid esters and about 1/10 for C10-ester. This can cause an important variation in the fruity scent of the wines. On the opposite, the relevant acids do not significantly variate.
Also the C6-alcohols seems to decrease proportionally with the proof lowering.
As important varietal compounds, mostly responsible of the floral scent above all in the case of Muscat wines, also a very little decrease roughly of 5-10% both in single compounds and in the sum could be estimated. This also for the 4-terpineol, monoterpenol connected to the intensity of the drying phase.
For the monoterpenols, we have a quite linear important decrease above all for linalool with an initial decrease and a subsequent increase for citronellol, nerol, and geraniol while α-terpineol tends always to increase. These different situation should be judged by considering that α-terpineol is also a chemical transformation compounds from linalool, nerol and geraniol, and that other variations can be justified with a longer time of the treatment, therefore favouring chemical hydrolysis of the bound forms and possible secondary metabolic reaction as that of citronellol yeast formation from nerol and geraniol.
Excluding the 2,7% dealcoholization data, the β-damascenone trend is slightly increasing due to the concentration effect of the technique and to the possible improved hydrolysis of the precursors.
The vinylphenols and benzaldehyde seems to have a strange evolution situation.

Industrial low dealcoholization (up to 2%) experiences on tipical Italian wines

We compared the variation among the wine that comes from industrial experience of limited Reduced Alcohol wine, with different alcohol removal degrees, as written in columns heading (Tab 3).
We observed a decrease from ethyl esters (iC5, C4-C10) similar among the wines varying between 60 and 87%, tendencially a few higher for the MW-higher esters. An exception is represented for the MW-lower esters in the case of Chianti, likely due to a limited fermentation by Brettanomyces in the dealcoholized sample favoured by the reduction of antiseptic effect of alcohol and by the possible airing occurred during the treatment.
Also the relevant acids presented a limited decrease in the case of Sfursat wines and Chianti (meanly at about 20%), but a rather constance or a few increase in the other cases.
As for the acetates – also important in contributing to the fruity scent -, both the most representing one, the isoamyl acetate, and the total sum of them have a level reduction of about 20%.

For the alcohols 2-phenylethanol, methionol and benzyl alcohol we can observe a small decrease from 5 to 20%, a little higher for methionol except for the Valpolicella wine with a possible alteration due to the Brett metabolism.
As aroma interesting trace-compounds grouped as 'phenols' like eugenol, guaiacol, o- and p-cresol and phenol, when properly evaluated as a sum in our wines, even a possible decrease of about 20% can be estimated. This also for the oak lactones, however with a quite constance in the case of Valpolicella wine.
In the case of C6-alcohols, a little decrease can also be observed at about 10%.
As important varietal compounds, a very little decrease roughly of 5-10% both in single compounds and in the sum could be estimated. This also for the 4-terpineol, monoterpenol connected to the intensity of the drying phase.
Finally we considered some norisoprenoids which are possible complementing scent in the wine just as young product, as the β-damascenone and β-ionone, or by progressed aging as TDN (1,1,3-trimethyl-1,2-dihydronaphthalene), vitispiranes (VTP), actinidols and relevant ethyl ethers. The relevant concentrations of the ethyl ethers are quite constant, while those of the compounds can have either an important decrease as for TDN until about 50% or a little more reduced variation as for VTP.
By concluding these comparison, the de-alcoholisation process limited to about 1.5 % proof could reduced the most important aroma influencing known volatiles in a similar way without a clear effect of the initial alcohol proof (please, consider the different level in the wine types considered). This reduction can variate between 5 and 30-40% with a mean of about 20%, seeming the range not depending of the wine types as from the results of the two similar Sfursat wines treated.

Sensory Analyses

The application of Friedman test revealed significant differences between divers dealcoholized rate wines for these descriptors: structure, persistence and sweet. We could note in fact a marked effect of alcohol on sensory perception of the structure and persistence, despite the increase of the extract and we can reconfirm also the important contribution of ethanol on the perception of sweet (Fig. 2).
We highlight relatively large differences, but not statistically significant, for descriptors acidity, salty and honey. However, the results of sensory analysis are sometimes controversial because they do not follow a progressive trend depending on the level of dealcoholization of the wines, so further investigations are required.

Conclusions

The testing performed and the data of industrial processes with membrane contactor can lead to the following conclusions:

- The alcohol membrane separation technique leads to a depletion of aroma compounds in the wines, especially for the most volatile like ethyl esters and lesser the acetates; these are the fermentative aromas.
- Concerning about the varietal aroma compounds it was observed a little decreasing trend on terpenic compounds; in the Norisoprenoid class the TDN presents a significant decreasing, while the β-damascenone data remains substantially unchanged.

- The de-alcoholisation process limited to about 1.5 % proof could reduced the most important aroma influencing known volatiles in a similar way without a clear effect of the initial alcohol proof (please, consider the different level in the considered wine types). This reduction can variate between 5 and 30-40% with a mean of about 20%, seeming the range not depending of the wine types as from the results of the two similar Sfursat wines treated.
- Therefore, any treatment with membrane contactor is recommended in young wines characterized by an high ester content; in case of varietal aromas wines the approach can be, knowing these results, varied depending on the volatile compounds that will characterize these wines.
- However, it is advisable to operate the dealcoholization treatment on wine fractions containing no aromatic substances.
- Finally, it should be technically noted that this technique helps to improve the tartaric stability; in contrast slight oxygenation and alcohol removal can promote the *Brettanomyces* development.
- In the dealcoholization of Moscato wines have been shown a substantial modification of the sensory profile: these wines are less structured, persistant and sweet depending on the different rates of dealcoholization; this aspect is very important to be considered in the production of wines which are well valued for the consumers.

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Figures

Fig. 1 - Functioning principle of "contactor"

Fig. 2 - Sensory profiles of dealcoholized wines (* *p* of significance < 0.05)

Tables

Tab. 1 - Main chemical characteristic of wines dealcoholized at different rates by a contactor membrane.

Tab. 2 - Aroma compounds of wines dealcoholized at different rates by a contactor membrane.

Tab. 3 - Aroma compounds of industrial dealcoholized (up to 2%) tipical Italian wines.

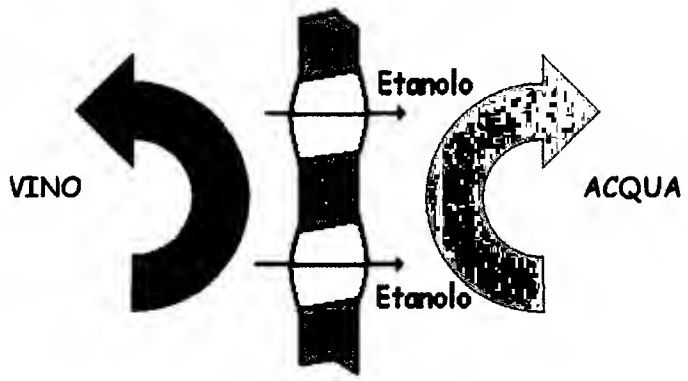

Fig. 1

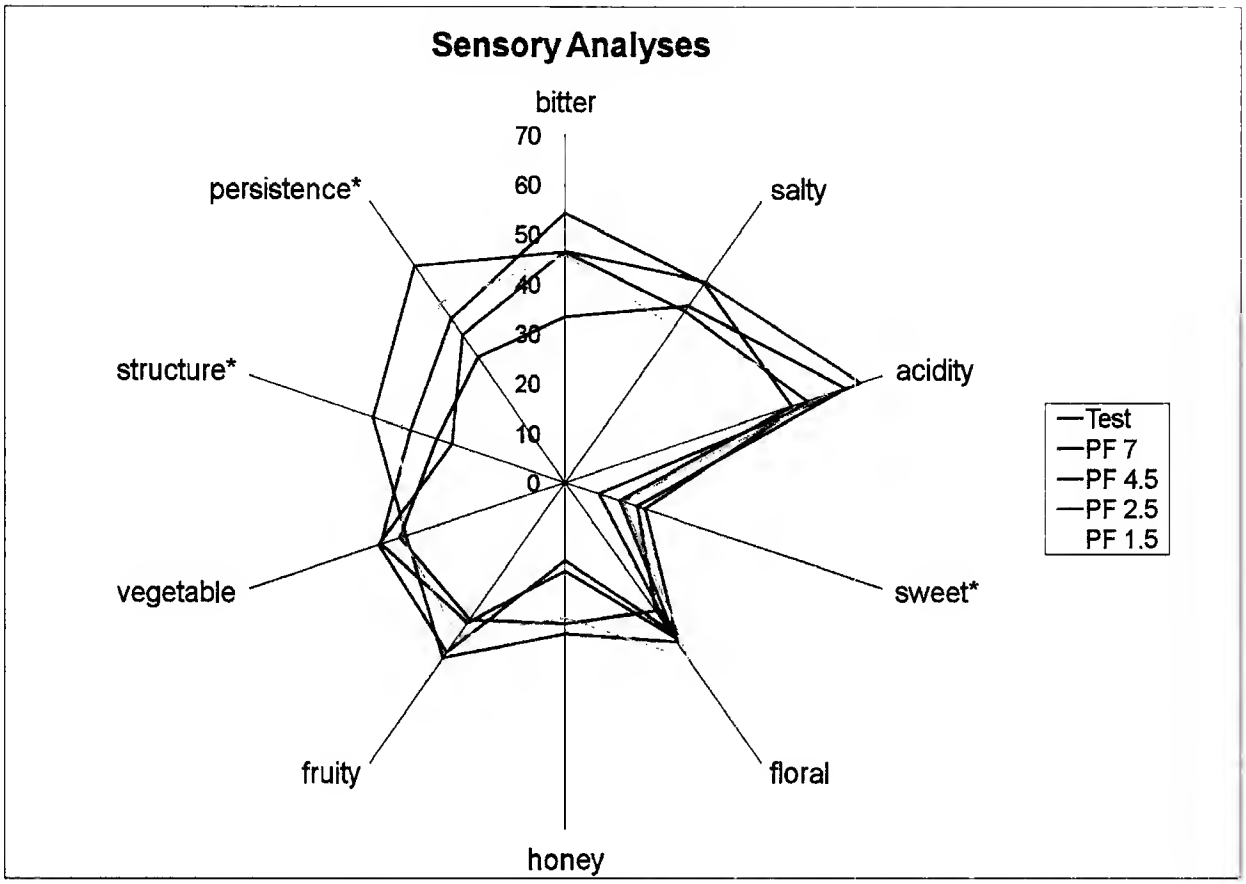

Fig. 2

% Alcohol removed		0 Test	1,4	2,7	5,1	7,1
Alcohol	**(% vol)**	12,4	11,0	9,7	7,3	5,3
pH		3,46	3,44	3,49	3,48	3,47
Titratable Acidity	**(g/L)**	6,6	6,6	6,7	6,7	6,9
Volatile Acidity	**(g/L)**	0,07	0,06	0,06	0,05	0,05
Acetic Acid	**(g/L)**	0,01	0,01	0,01	0,01	0,01
Total exctract	**(g/L)**	23,5	23,9	23,2	24,2	24,7
Glycerol	**(g/L)**	10,1	10,8	10,8	11,1	11,5
Tartaric acid	**(g/L)**	1,82	1,79	1,90	1,88	1,89
K	**(mg/L)**	638	664	653	675	709
Ash	**(g/L)**	1,89	1,80	1,81	1,81	2,17
Ash alkalinity	**(meq/L)**	20,23	18,8	19,0	21,0	21,3
Saturation temperature	**(°C)**	22,1	20,4	20,1	17,8	16,5

Tab. 1

COMPOUND CLASS	COMPOUNDS TESTED FOR SPME	Test	PF 1,5	PF 2,5	PF 4,5	PF 7
Acetates	Hexyl acetate	240,9	199,6	161,5	100,0	86,6
	Isoamyl acetate	746,8	642,6	516,2	349,4	289,5
	Ethyl acetate	398,4	285,9	201,2	165,6	126,6
	SUM ACETATE	1386,1	1128,2	878,8	615,0	502,7
Ethyl esters	Ethyl butyrate (C4 ethyl)	55,7	44,8	35,8	23,6	20,1
	Ethyl caproate (C6 ethyl)	701,3	546,4	450,9	271,6	230,7
	Ethyl caprylate (C8 ethyl)	4762,3	3485,7	2815,9	1727,8	1136,4
	Ethyl Caprino (C10-ethyl)	4953,3	3846,5	2723,0	1701,3	743,7
	Ethyl-2-methyl-butanoate	1,0	0,8	0,6	0,5	0,4
	Ethyl-3-methyl-butanoate	0,8	0,8	0,6	0,5	0,4
	SUM ETHYL ESTERS	10474,5	7925,0	6026,9	3725,2	2131,8
C6 alcohols	Hexanol	168,7	172,2	116,2	108,2	72,1
	Trans-3-hexenol	4,3	5,0	3,4	3,0	2,1
	Cis-3-hexenol	28,4	32,4	17,7	19,0	13,6
	SUM ALCOHOLS C6	201,4	209,5	137,4	130,3	87,8
Alcohol various	Beta-Phenyl Ethyl Alcohol	12071,2	11242,7	6867,5	8560,2	6984,6
Terpene alcohols	Linalool	2895,3	2566,0	1885,4	2065,3	1707,3
	HO-Trienolo	262,5	354,3	172,5	476,6	528,2
	Alpha-Terpineol	329,8	472,1	208,7	627,7	688,1
	Citronellol after 1-Decanol	25,9	23,1	16,4	16,0	10,7
	Nerol under 4-Hydroxy Ethyl butyrate	36,4	44,9	25,0	55,4	52,4
	Geraniol ethyl below laurina	209,0	166,3	85,0	115,4	100,8
	D-Limonene	154,9	172,9	56,8	103,4	93,9
	SUM terpene alcohols	3913,7	3799,6	2449,9	3459,7	3181,4
Oxides of linalool	Trans-linalool oxide (C) (Pir)	24,4	29,0	20,2	30,6	29,2
	Cis-Rose oxide under hexanol	3,3	3,4	1,9	1,6	1,3
	Trans-Rose Oxide	0,7	0,6	0,4	0,2	0,2
	SUM linalool oxide	28,4	33,0	22,4	32,4	30,7
Terpene ethers	Linalyl Ethyl ether	479,0	534,8	160,3	249,5	172,6
	Neryl Ethyl ether	12,3	13,1	4,4	7,2	5,3
	Geranyl ethyl ether	259,3	275,6	15,0	6,0	449,8
	Alfa Terpenil Ethyl ether	12,4	14,4	10,5	15,7	14,8
	SUM ETHERS Terpenes	762,9	837,9	190,0	278,4	642,5
Vinylphenol	4-Vinyl-Guaiacol	40,7	28,4	26,1	29,5	26,3
	4 Vinyl Phenol	0,0	14,1	11,6	14,4	13,1
	SUM vinylphenol	40,7	42,5	37,7	43,9	39,4
Aromatic aldehydes	Benzaldehyde	12,1	34,0	19,0	30,3	25,2
Fatty acids	Butyric	15,7	18,2	12,7	14,9	12,6
	Caprylic acid (C 8)	2075,5	2262,0	1716,5	2185,6	2010,5
	SUM FATTY	2091,2	2280,3	1729,2	2200,5	2023,1
Norisoprenoids	Beta Damascenone SOT-dop Cinammato E	19,4	22,9	11,8	30,2	31,6
	Beta-Ionone	0,0	0,0	0,0	0,0	0,0

Tab. 2

		Sfurzat		Sfurzat		Chianti		Valpolicella	
COMPOUND CLASS	**COMPOUNDS TESTED BY SPME**	**16,6 %v/v**	**-1,7 %v/v**	**15,9 %v/v**	**-1,3 %v/v**	**13,9 %v/v**	**-1,0 %v/v**	**14%v/v**	**-1,7 %v/v**
Acetates	Hexyl acetate	4,3	3,8	13,6	14,5	8,8	5,1	21,4	12,6
	Isobutyl acetate	2,8	2,2	2,4	2,2	1,3	1,0	2,8	0,9
	Isoamyl acetate	79,4	71,5	161,9	147,1	107,6	83,3	339,6	204,0
	Beta-phenylethyl acetate	102,1	42,0	41,9	33,2	47,3	42,6	38,1	32,2
	Ethylphenyl acetate	7,3	5,4	4,5	4,5	4,9	4,8	2,4	2,2
	SUM ACETATE	196,0	124,9	224,4	201,5	169,8	136,7	404,3	251,9
Ethyl esters	Ethyl butyrate (C4 ethyl)	33,9	26,8	31,1	27,2	16,5	14,0	42,2	26,7
	Ethyl caproate (C6 ethyl)	327,7	294,6	350,3	320,7	240,9	222,1	493,0	342,0
	Ethyl caprylate (C8 ethyl)	1.400,1	862,2	1.070,3	945,9	1.018,3	1.345,8	2.163,6	1.639,5
	Ethyl Caprino (C10-ethyl)	783,5	343,3	537,5	431,2	731,6	946,4	742,8	513,0
	Ethyl-2-methyl-butanoate	6,6	5,9	6,1	5,2	5,6	5,7	1,7	1,2
	Ethyl-3-methyl-butanoate	10,5	9,1	8,9	7,8	6,8	6,0	2,9	2,1
	SUM ETHYL ESTERS	2.562,3	1.541,8	2.004,3	1.738,0	2.019,7	2.539,9	3.446,1	2.524,5
C6 alcohols	Hexanol	696,4	619,3	856,0	672,7	360,6	323,4	247,7	214,5
	Trans-3-hexenol	9,7	7,6	8,8	6,7	9,0	7,1	4,3	3,4
	Cis-3-hexenol	20,0	12,7	16,9	11,8	10,7	10,3	11,0	7,5
	2-Hexen-1-ol	1,4	1,3	1,5	1,0	0,9	0,7	0,6	0,4
	SUM ALCOHOLS C6	727,5	640,9	883,2	692,2	381,2	341,5	263,6	225,8
Various alcohols	Benzyl alcohol	49,8	37,6	33,1	22,8	57,4	55,6	13,7	11,5
	Beta-Phenyl Ethyl Alcohol	8.318,6	5.442,0	7.582,2	5.304,0	7.095,8	6.745,8	2.949,9	2.443,3
	3-Methyl-thio-propanol (Methionol)	36,8	28,5	42,2	25,1	64,0	69,2	28,6	23,7
	SUM VARIOUS ALCOHOLS	8405,2	5508,1	7657,5	5351,9	7.217,2	6870,6	2992,2	2478,5
Terpene alcohols	Linalool	32,8	32,4	28,9	24,4	44,5	42,4	141,0	155,2
	HO-Trienolo	4,1	2,2	1,8	1,6	0,4	0,3	0,7	0,7
	Alpha-Terpineol	18,4	15,5	13,1	10,5	9,8	9,0	34,4	38,2
	Citronellol after 1-Decanol	11,9	10,3	12,3	10,7	21,7	20,9	20,2	21,4
	Nerol under 4-Hydroxy Ethyl butyrate	3,9	2,6	3,9	3,1	3,0	3,5	4,3	4,8
	Geraniol ethyl below laurina	3,8	2,8	3,4	2,6	6,6	5,8	7,6	8,9
	D-Limonene	9,2	3,5	7,1	3,6	3,8	3,1	20,8	14,7
	4-Terpineol	103,3	89,8	143,7	115,7	2,8	2,7	2,0	1,8
	SUM terpene alcohols	187,5	159,0	214,2	172,3	92,7	87,6	231,0	245,8
Ethylphenol	4-Ethyl Phenol	18,6	14,1	13,3	10,8	19,8	59,8	3,8	24,3
	4 - Ethyl Guaiacol	4,7	3,6	5,7	4,6	5,9	17,5	1,0	21,1
Phenols varietal	Eugenol	7,2	5,0	3,3	3,0	1,5	1,4	3,6	3,8
	Guaiacol	4,8	3,5	3,3	2,3	0,9	0,8	1,3	1,2
	Ortho-Cresol	1,9	1,0	1,4	0,8	0,9	0,7	1,1	0,8
	Paracresol	2,7	1,5	1,9	1,5	0,8	1,0	1,0	0,7
	Phenol	7,6	4,7	6,8	4,7	3,4	2,9	7,9	6,6
	SUM PHENOLS VARIETY	24,2	15,7	16,7	12,4	7,5	6,9	14,9	13,2

		Sfurzat		Sfurzat		Chianti		Valpolicella	
COMPOUND CLASS	**COMPOUNDS TESTED BY SPME**	**16,6 %v/v**	**-1,7 %v/v**	**15,9 %v/v**	**-1,3 %v/v**	**13,9 %v/v**	**-1,0 %v/v**	**14%v/v**	**-1,7 %v/v**
Aromatic aldehydes	Phenyl Acetaldehyde	59,5	36,1	33,2	21,9	26,0	23,3	17,2	11,8
	Benzaldehyde	135,2	115,5	117,7	91,3	39,2	17,9	31,9	10,4
	SUM aromatic aldehydes	194,6	151,6	150,9	113,2	65,2	41,2	49,1	22,2
Oak Lactones	Oak Lattone 1 (Whiskey lactone)	30,5	23,0	11,9	9,4	2,2	2,1	0,8	0,0
	Oak Lattone 2 (Whiskey lactone)	51,6	38,9	23,6	19,5	2,6	2,8	0,0	0,0
	SUM OAK Latton	82,1	62,0	35,5	29,0	4,8	4,9	0,8	0,0
Fatty acids	Butyric	21,2	15,1	21,0	14,6	9,5	8,6	20,9	18,3
	Ac Iso-Valeriani	33,1	27,1	34,1	27,0	53,4	51,6	34,4	30,5
	Caproic acid (C 6)	316,6	234,3	276,6	205,0	160,0	165,9	275,8	259,9
	Caprylic acid (C 8)	1.009,5	629,3	665,0	527,6	543,5	505,1	835,8	896,0
	Capric acid (C 10)	408,6	208,8	229,8	186,7	484,4	273,7	242,6	269,3
	SUM free fatty	1.789,0	1.114,6	1.226,6	960,9	1.250,9	1.004,9	1.409,5	1.474,0
Norisoprenoids	Beta Damascenone SOT-dop Cinammato E	12,0	10,2	19,5	17,1	15,6	14,5	36,9	39,2
	Alpha-Ionone	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
	Beta-Ionone	1,0	0,8	0,8	0,5	0,5	0,5	0,5	0,4
	TDN	50,9	23,6	50,5	29,5	12,4	9,9	10,6	7,6
	Ethoxy TDN	2,5	2,1	2,4	2,3	1,2	1,5	1,1	1,3
	Vitispirano 1	305,1	278,0	341,7	265,1	48,3	48,9	86,3	77,9
	Vitispirano 2	103,1	91,3	115,0	94,4	25,3	25,6	57,7	49,1
	Actinidoli Ethoxy Derivatives 1	17,9	15,8	14,2	12,8	3,8	4,0	1,6	2,1
	Actinidoli Ethoxy Derivatives 2	11,4	9,8	8,5	8,0	2,5	2,5	1,1	1,4
	Actinidoli 1	3,3	2,6	2,4	1,9	0,9	1,2	0,6	0,7
	Actinidoli 2	5,5	3,8	4,5	3,7	2,0	2,1	1,2	1,3
	SUM NORISOPRENOIDS	512,6	437,8	559,6	435,1	112,4	110,8	197,6	181,1

Tab. 3

INTERVITIS INTERFRUCTA 2010

International technology trade fair for wine, fruit, fruit juice and spirits

Meny years' experiences on the dealcoholization processes using membrane techniques to guarantee the quality and sustainability of the wine

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Why to dealcoholize?

- Social reasons
- Health reasons
- Due to climate change

Climatic changes and the increasing of alcohol content in wines: a global emergency

- The Australian Wine Research Institute found that the average content of alcohol in Australian wines increase from 12.4% to 14% from 1984 to 2004 (average of 15% Shiraz)
- United States ---------->
- Similar trend for other producer countries

(2) ALCOHOL & SUGAR LEVELS IN CALIFORNIA
From CA Grape Crush Report District 4 – All varieties

Year	Alcohol
1971	12.5%
1973	12.7%
1975	13.2%
1976	13.7%
1978	14.0%
1981	13.7%
1983	13.1%
1986	13.3%
1990	13.5%
1992	13.8%
1997	14.4%
1999	14.6%
2001	14.8%

Extracted from Conibear (2006)

Reduction of the wines alcohol content

- Production of grapes with lower sugar content, but ”ripe"
  - Agronomic practices
    - Increase the yield
    - Still need to produce ripe and "balanced” grapes
  - Direct action on the grape-vine (selection, GMOs ...)
- Reduction of alcohol yield during the alcoholic fermentation
  - Low alcohol producers yeasts
- Changing the composition of musts or wines (decrease the concentration of sugars or alcohol)
  - Dilute the musts (and wine treated with RO)
  - Reduce sugar content of musts
  - Wine dealcoholization

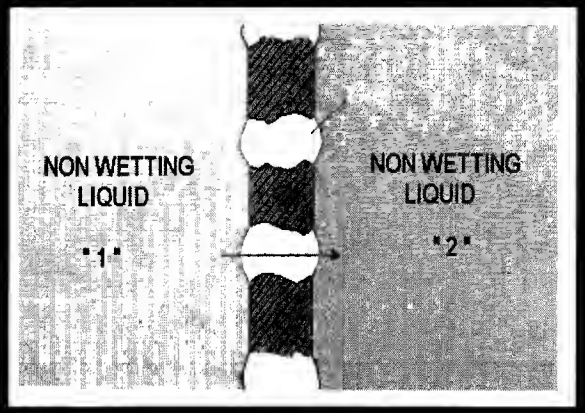
Wine dealcoholization methods

- Physical techniques directly on wine: Spinning Cone Column, runoff evaporation at atmospheric pressure, vacuum evaporation, supercritical extraction of alcohol with CO_2, other physical techniques.
- Membrane processes in combination with other techniques

Membrane technologies

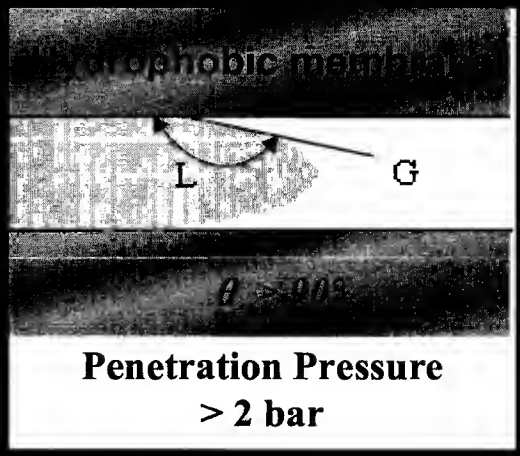
- RO + Distillation
- RO + Water
- ***"Contactor" membrane***
- ***Combination of membrane techniques***
- ***Pervaporation***

Gasseous Contactor Membrane



Principle

- *Hydrophobic membranes*
- *Operating P < Penetration P*
- *Makes contact between two liquids keeping them separated by a layer of gas*



Applications

- *Osmotic Distillation*
- ***Separation by gasseous membrane***

Contactors application in wines treatment: decreasing alcohol content (gas membrane separation)

- Driving force is a Δ in vapour pressure created by a Δ in concentration
- Extractant: water

$$N_{eth} = K_{m\,eth} \cdot \frac{P_{eth\,1} - P_{eth\,2}}{P_{A\,mt}}$$

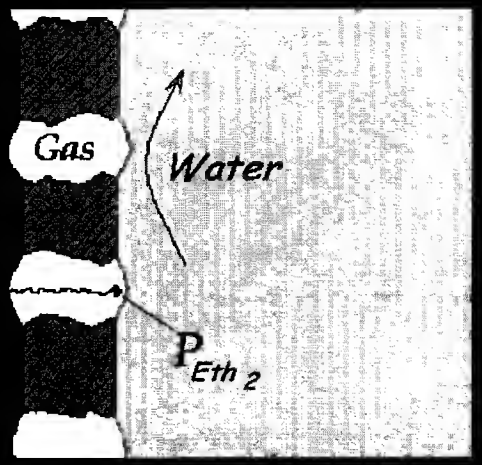
N_{eth} = *ethanol molar flow*

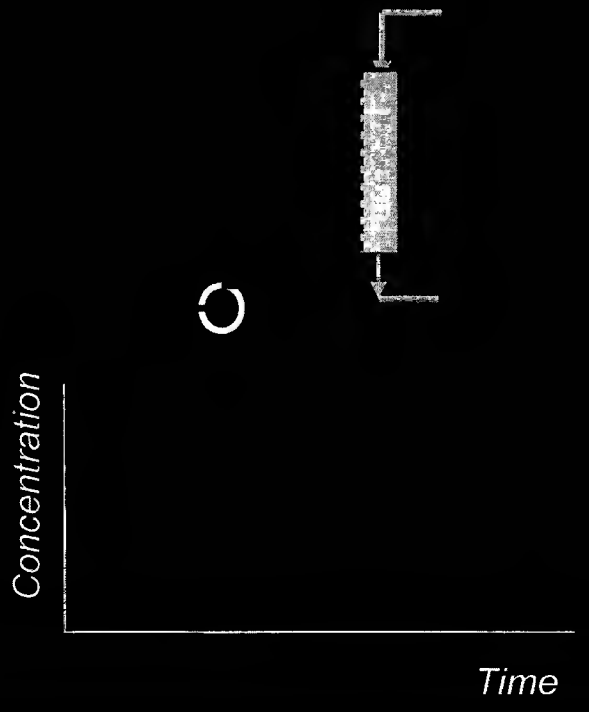
P_{eth1} = *ethanol vapour pressure in the wine*

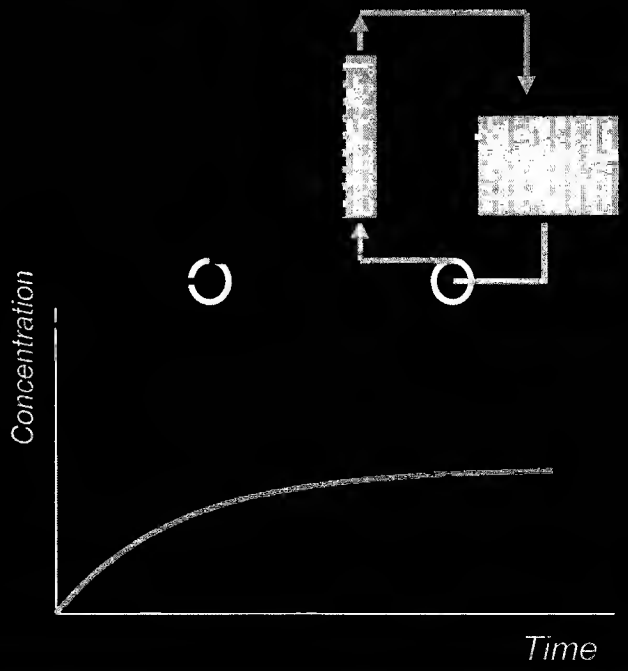
P_{eth2} = *ethanol vapour pressure in the extractant*

K_{meth} = *Diffusional permeability* = $K_{mw}\, D_{eth}/D_{WA}$

Alcohol reduction on wine



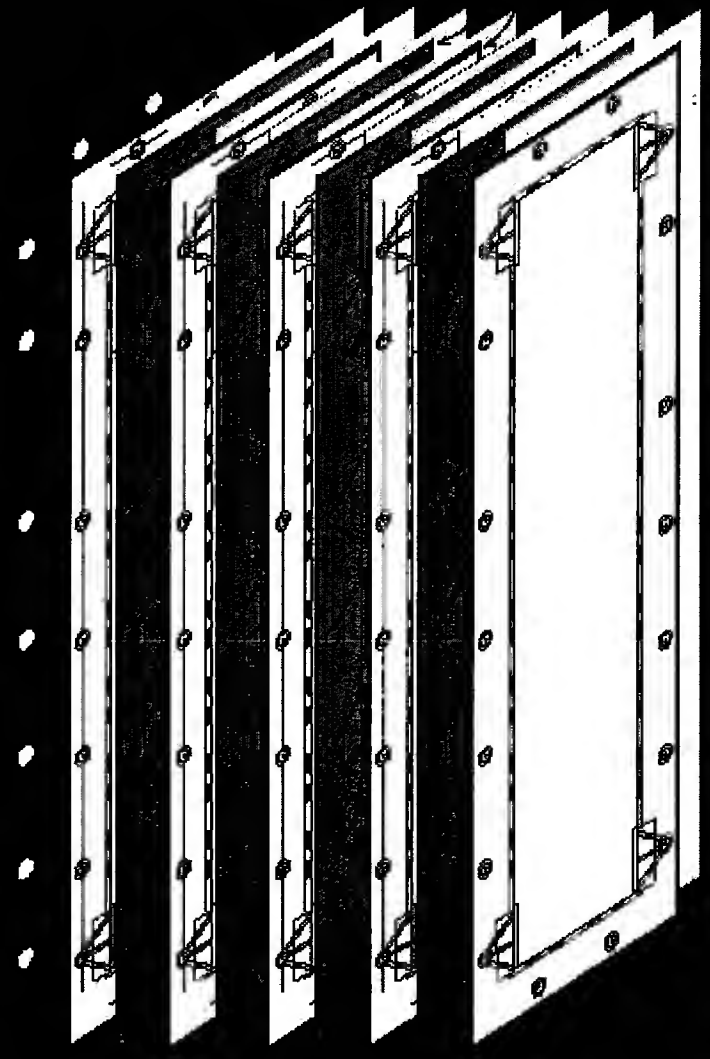




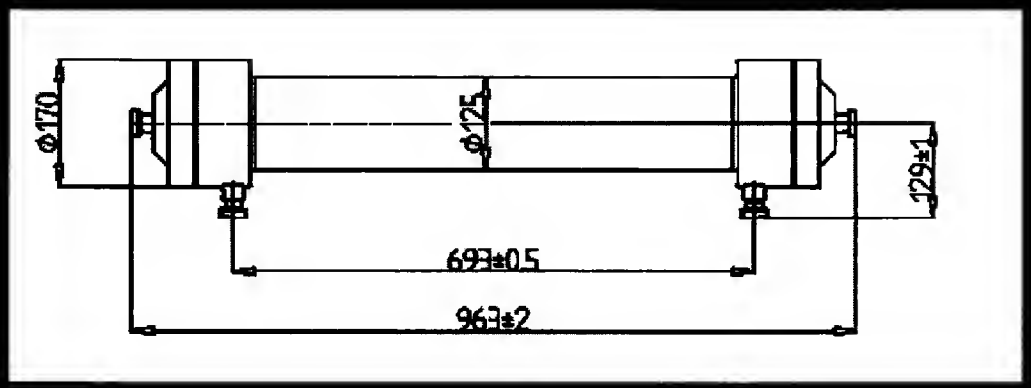
Contactor Membrane

PILOT PLANT

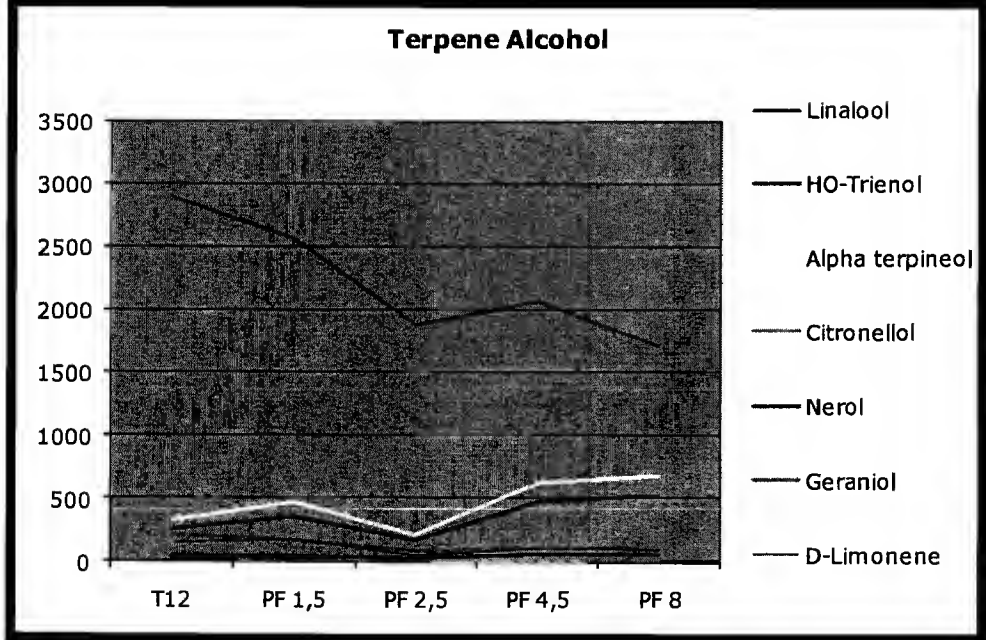
FRAME AND PLATE

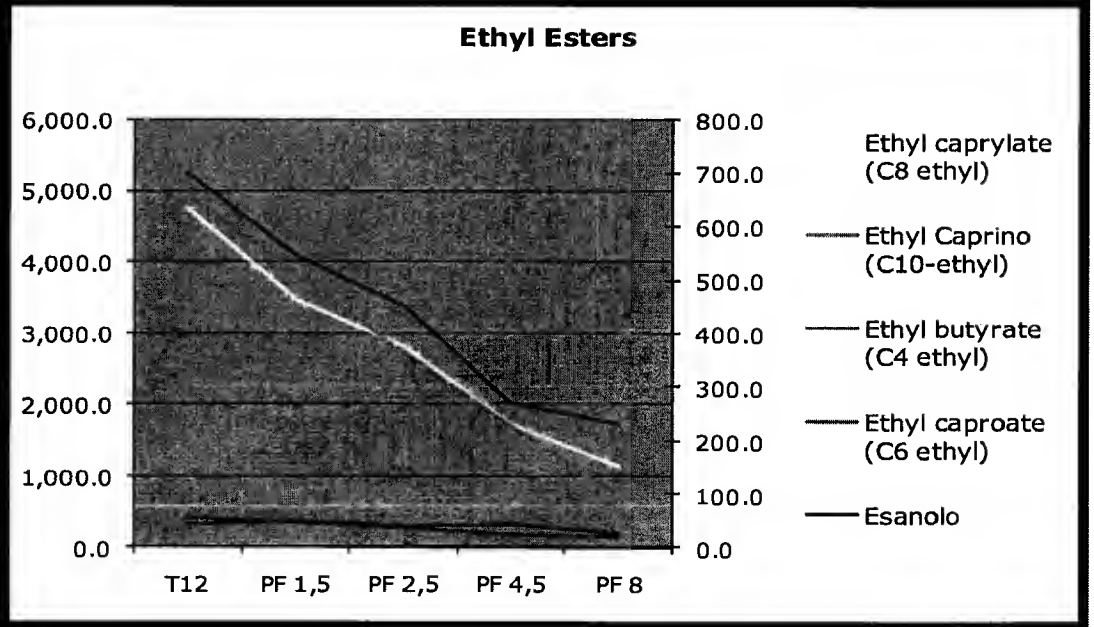


HOLLOW FIBER



Study of the effects of the dealcoholization rate with contactor membrane on the wine composition





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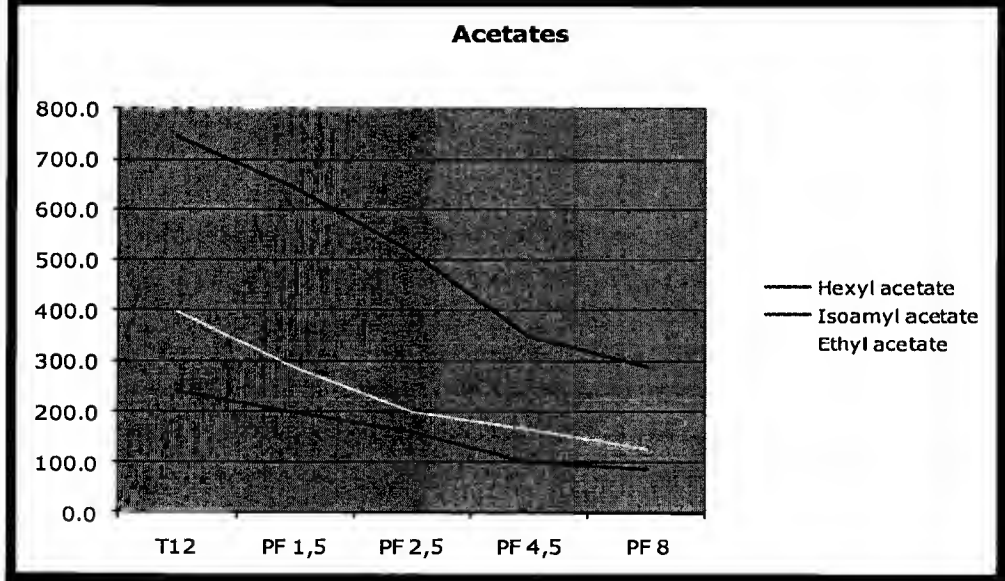
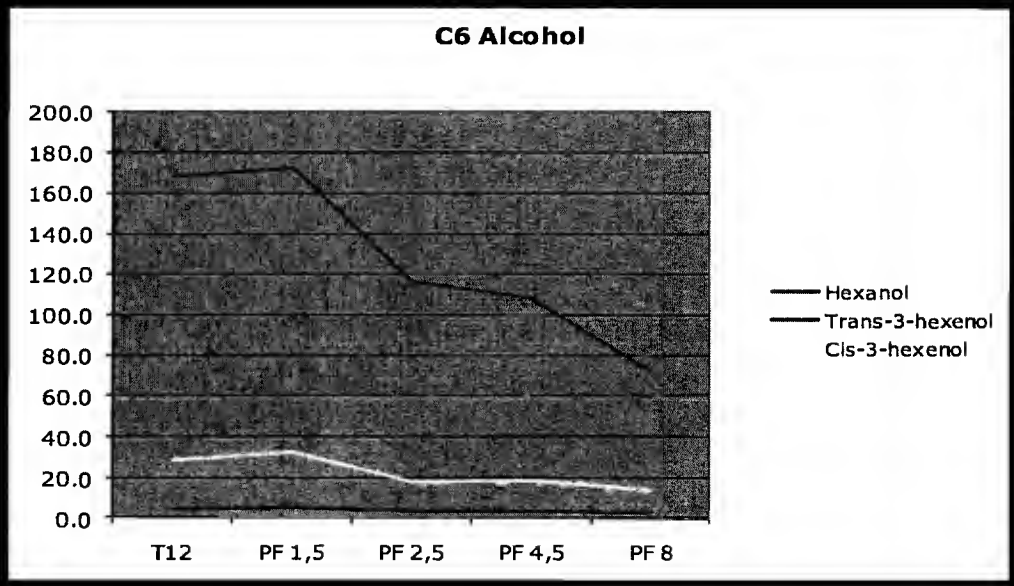
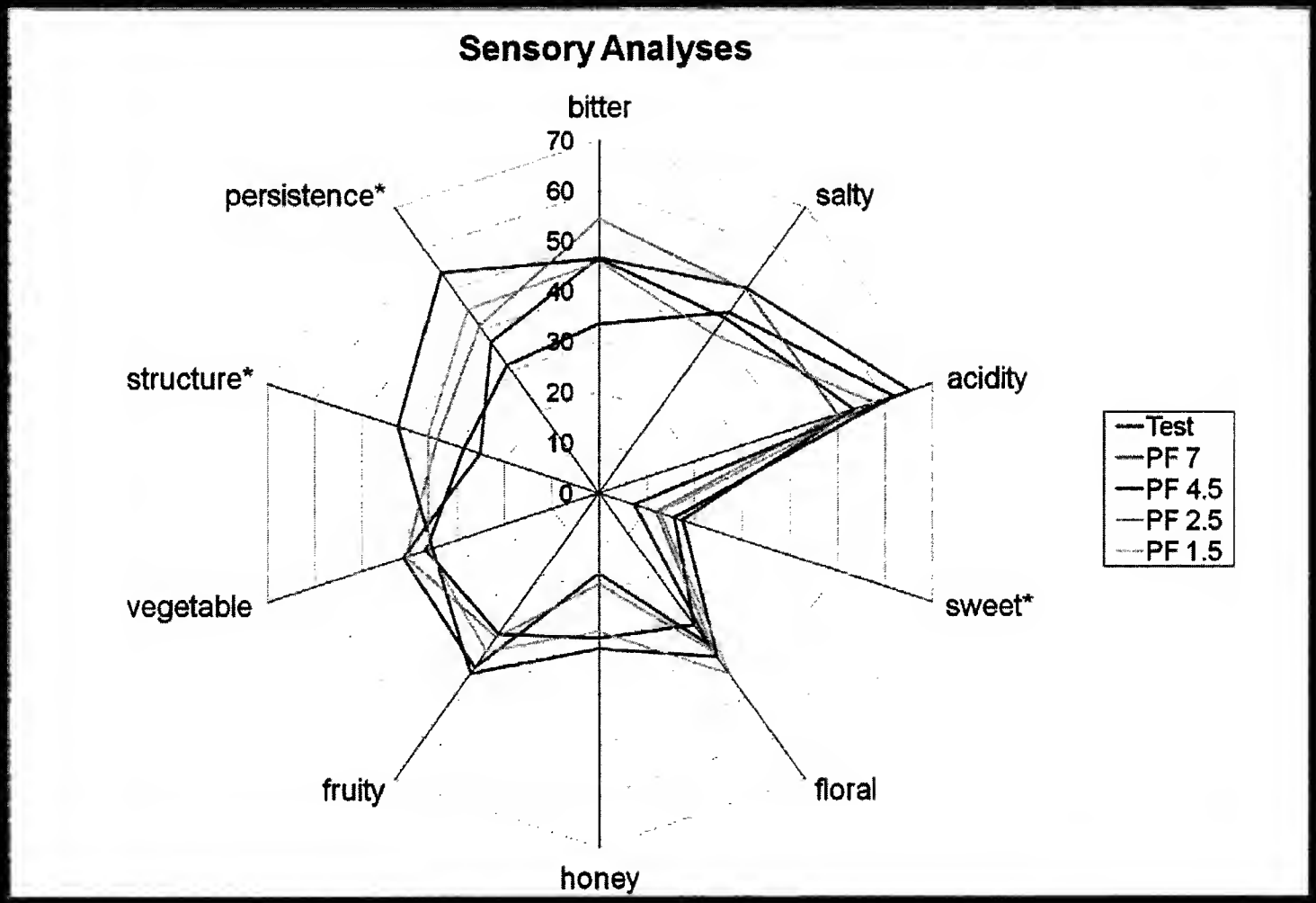




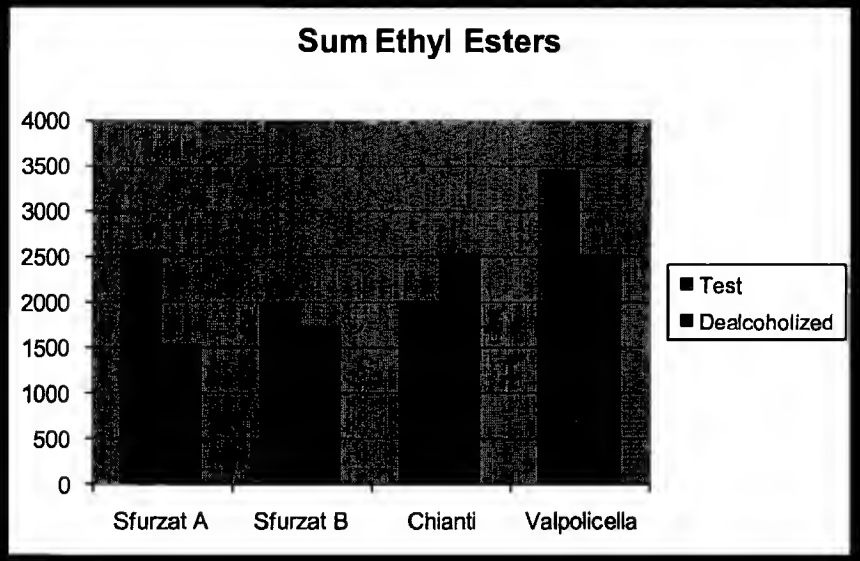
Study of the effects of the dealcoholization rate with contactor membrane on the wine composition

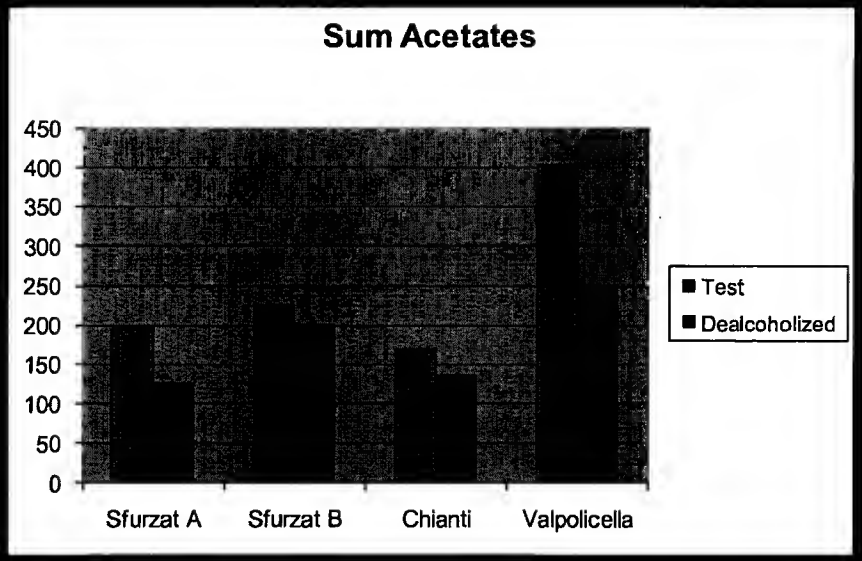
% Alcohol removed		0 Test	1,4	2,7	5,1	7,1
Alcohol	**(% vol)**	12,4	11,0	9,7	7,3	5,3
pH		3,46	3,44	3,49	3,48	3,47
Titratable Acidity	**(g/L)**	6,6	6,6	6,7	6,7	6,9
Volatile Acidity	**(g/L)**	0,07	0,06	0,06	0,05	0,05
Acetic Acid	**(g/L)**	0,01	0,01	0,01	0,01	0,01
Total exctract	**(g/L)**	23,5	23,9	23,2	24,2	24,7
Glycerol	**(g/L)**	10,1	10,8	10,8	11,1	11,5
Tartaric acid	**(g/L)**	1,82	1,79	1,90	1,88	1,89
K	**(mg/L)**	638	664	653	675	709
Ash	**(g/L)**	1,89	1,80	1,81	1,81	2,17
Ash alkalinity	**(meq/L)**	20,23	18,8	19,0	21,0	21,3
Saturation temperature	**(°C)**	22,1	20,4	20,1	17,8	16,5

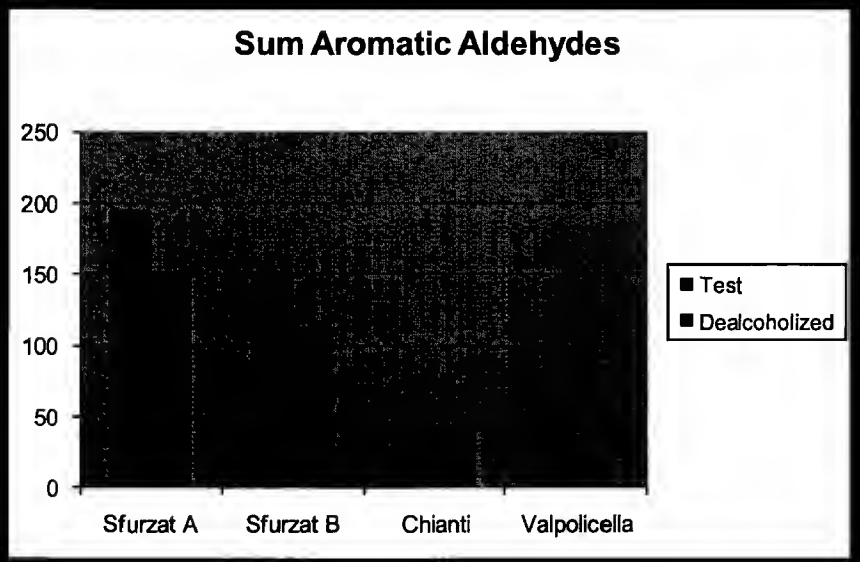
Sensory profile of dealcoholized Moscato

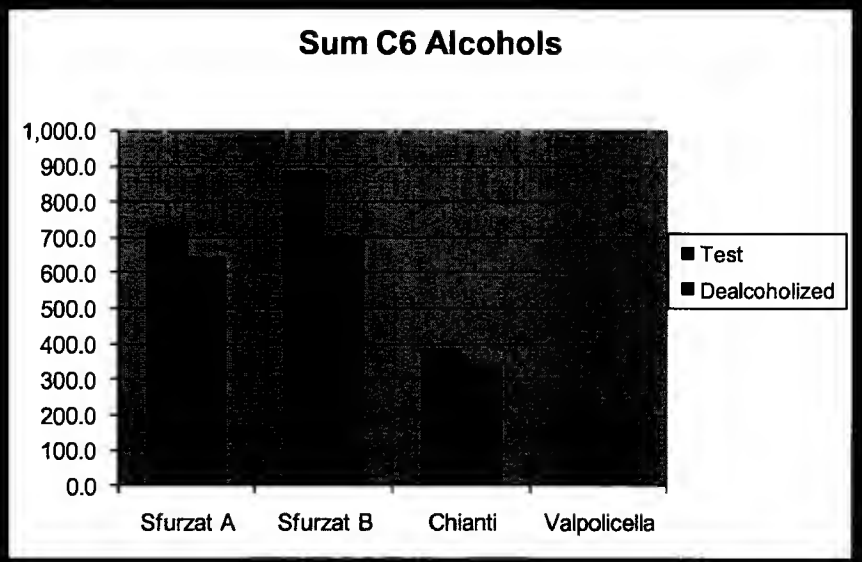


Industrial low dealcoholization (up to 2%) experiences on typical Italian wines

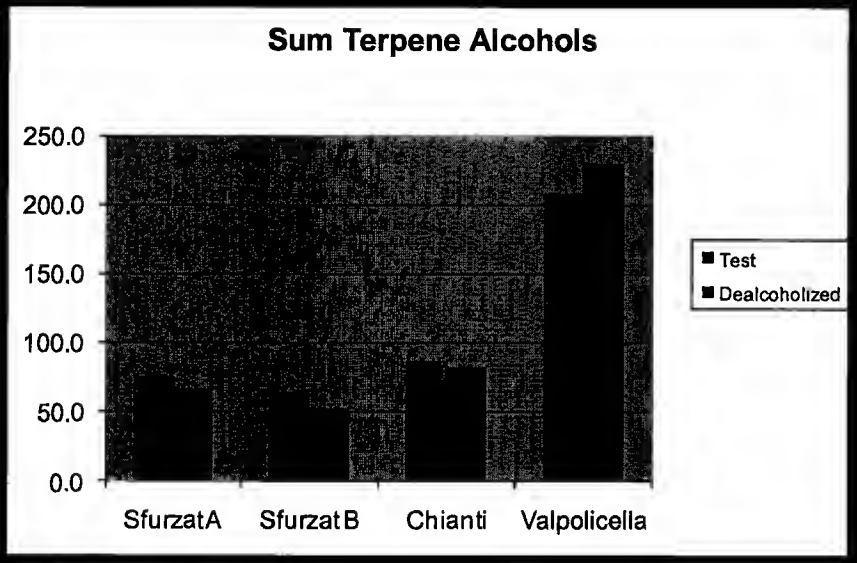


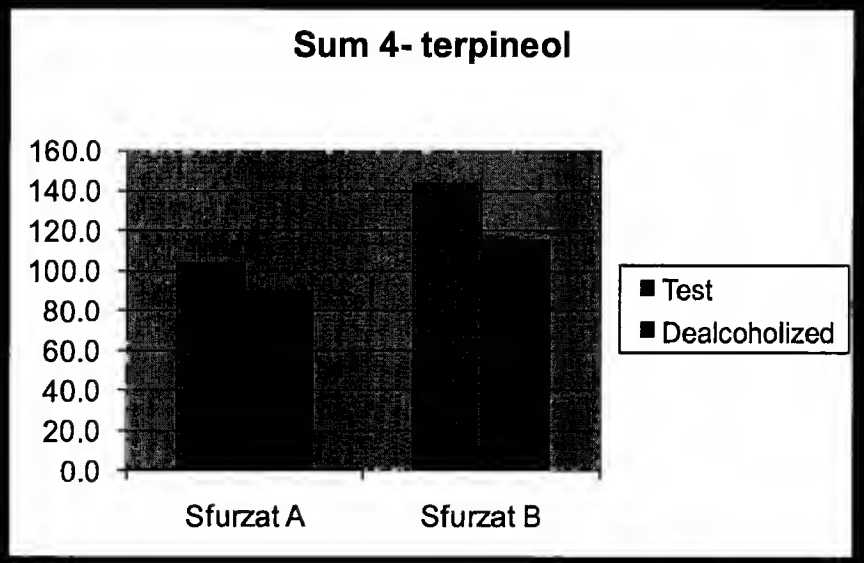


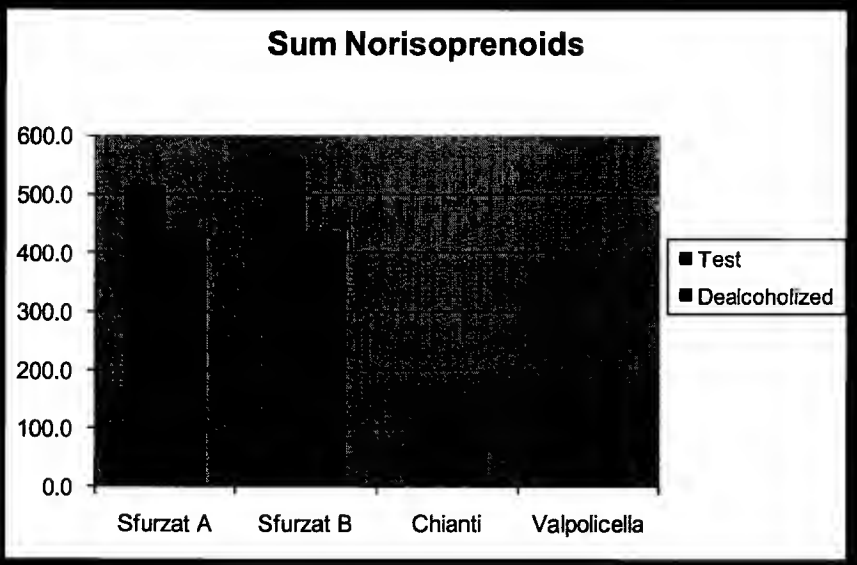


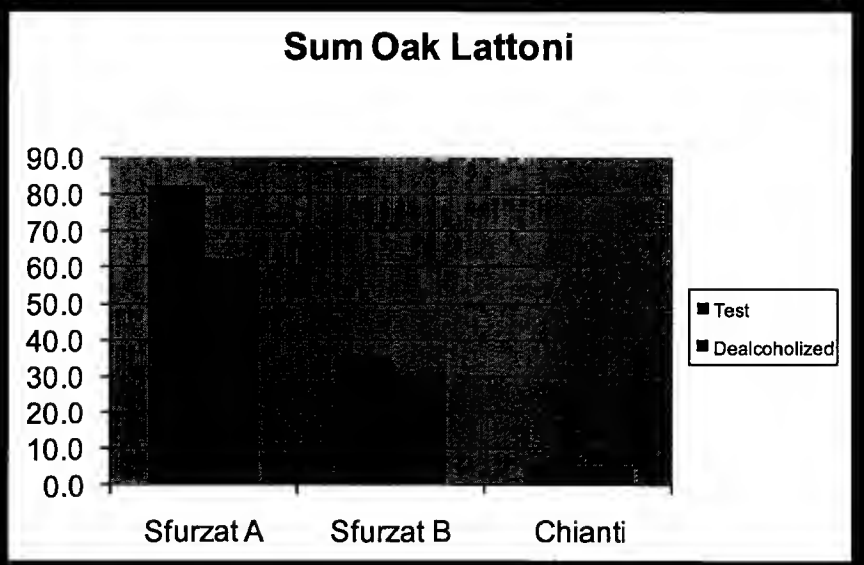


Industrial low dealcoholization (up to 2%) experiences on typical Italian wines

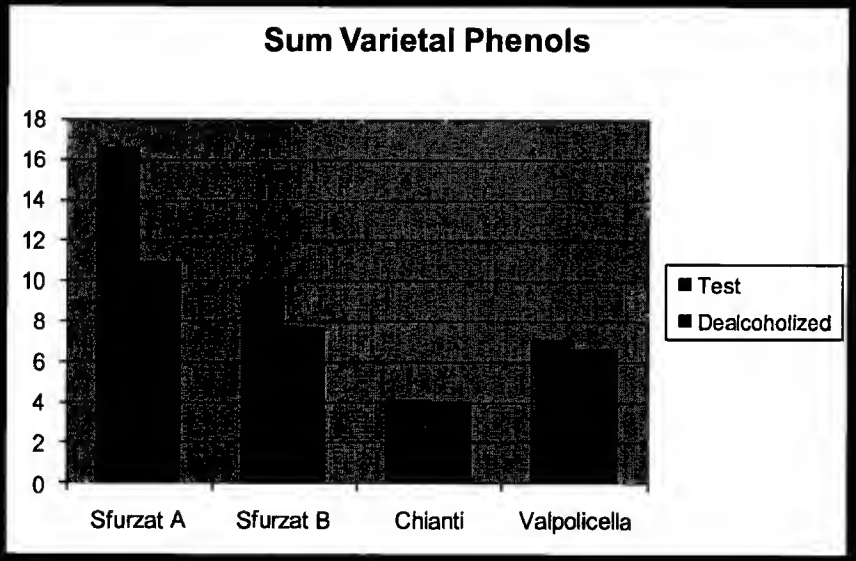


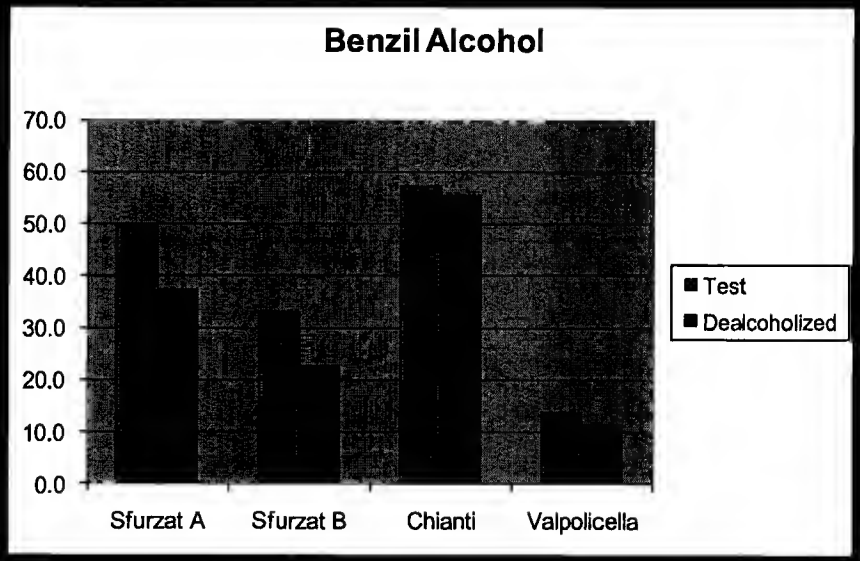


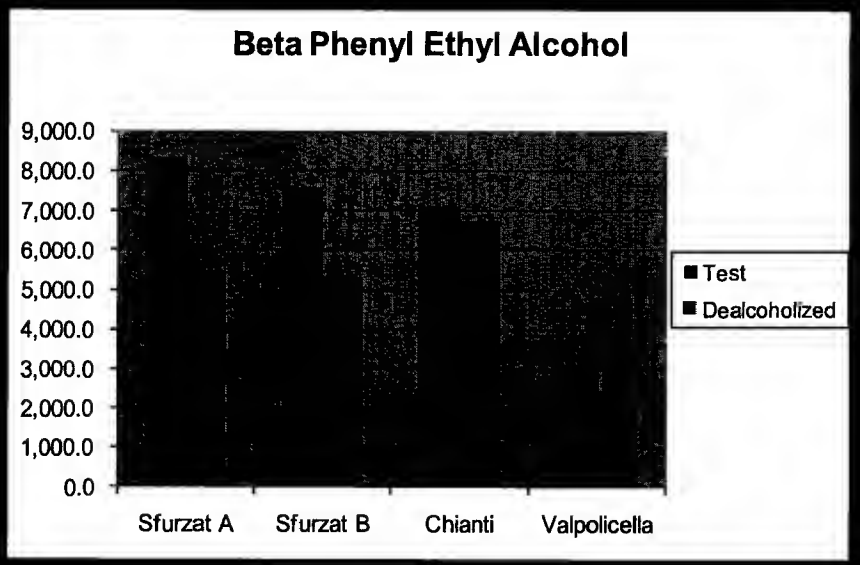


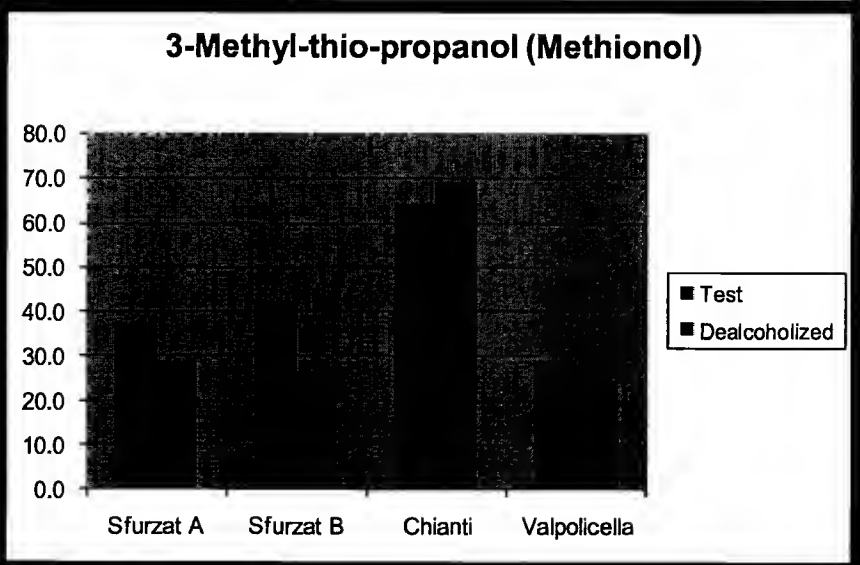


Industrial low dealcoholization (up to 2%) experiences on typical Italian wines

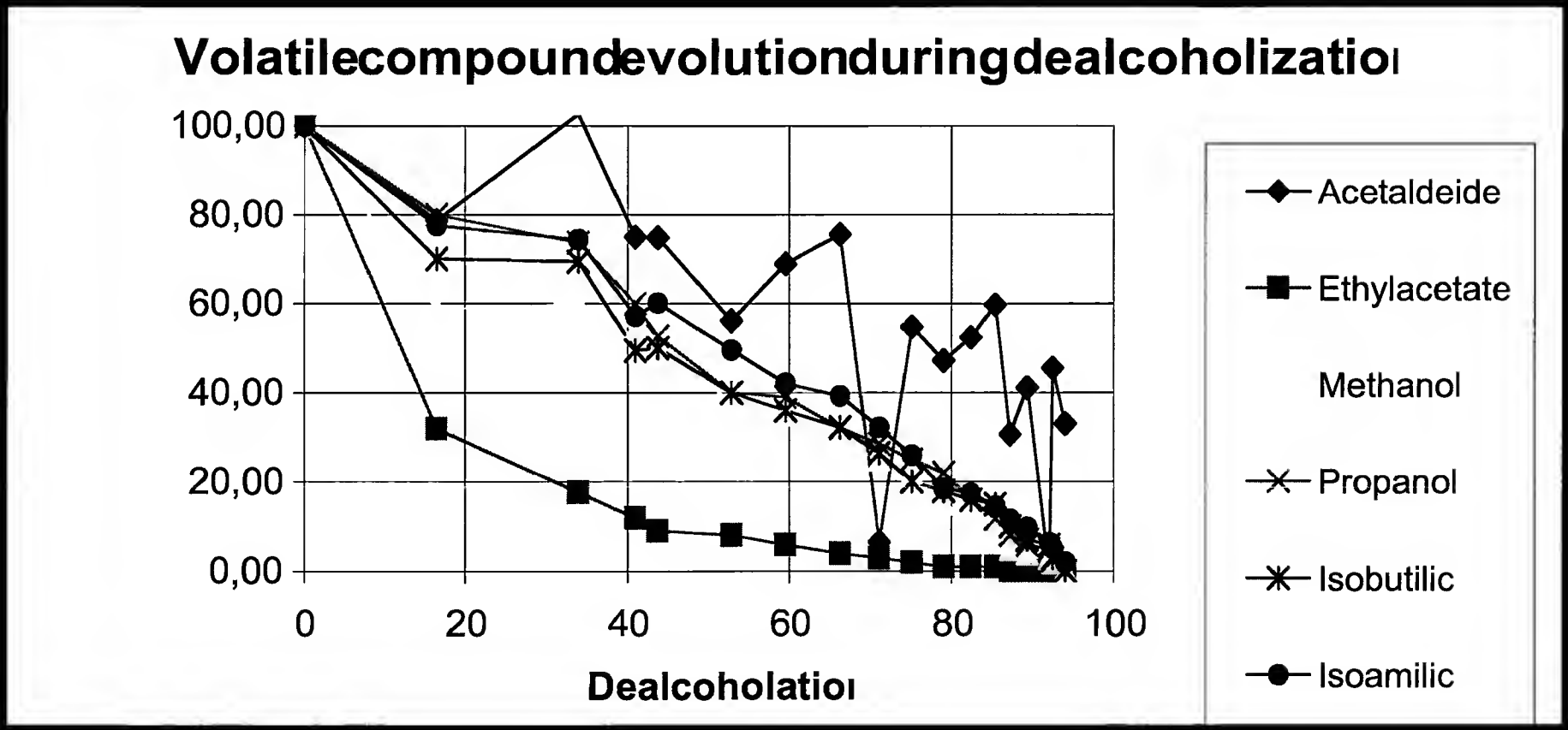








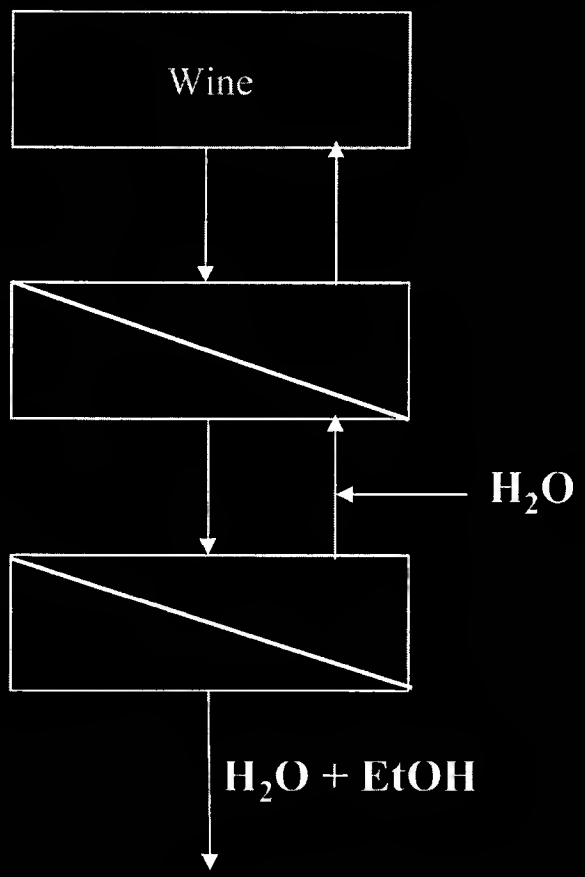
Other volatile compounds removed during dealcoholization process

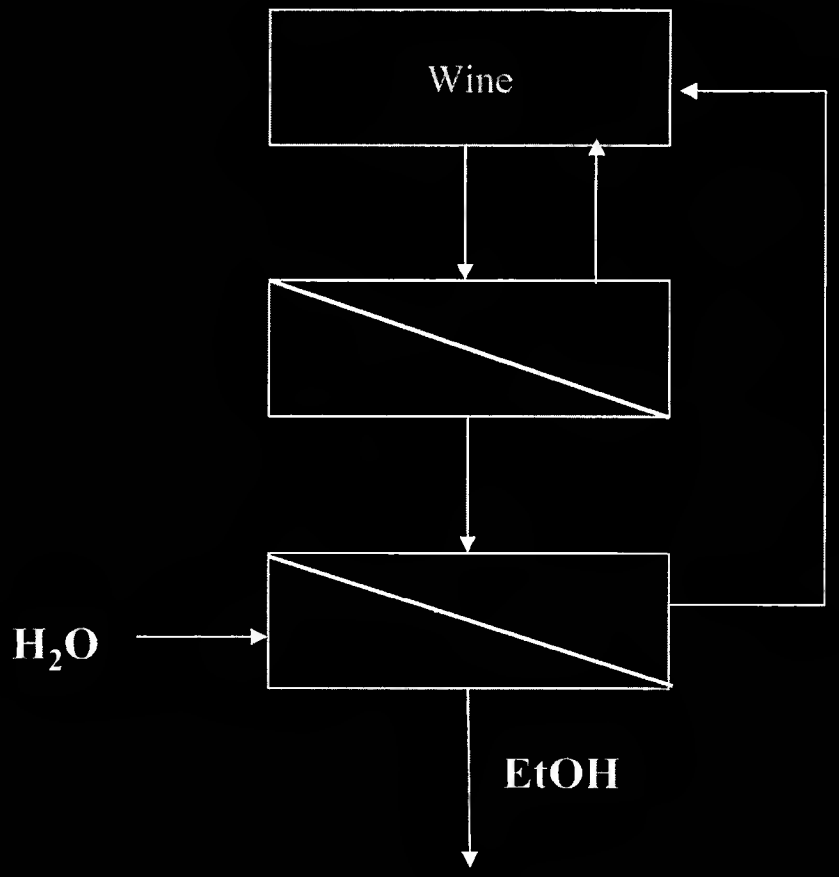


OTHER SCHEMES FOR DEALCOHOLIZATION COMBINED WITH MEMBRANE

1

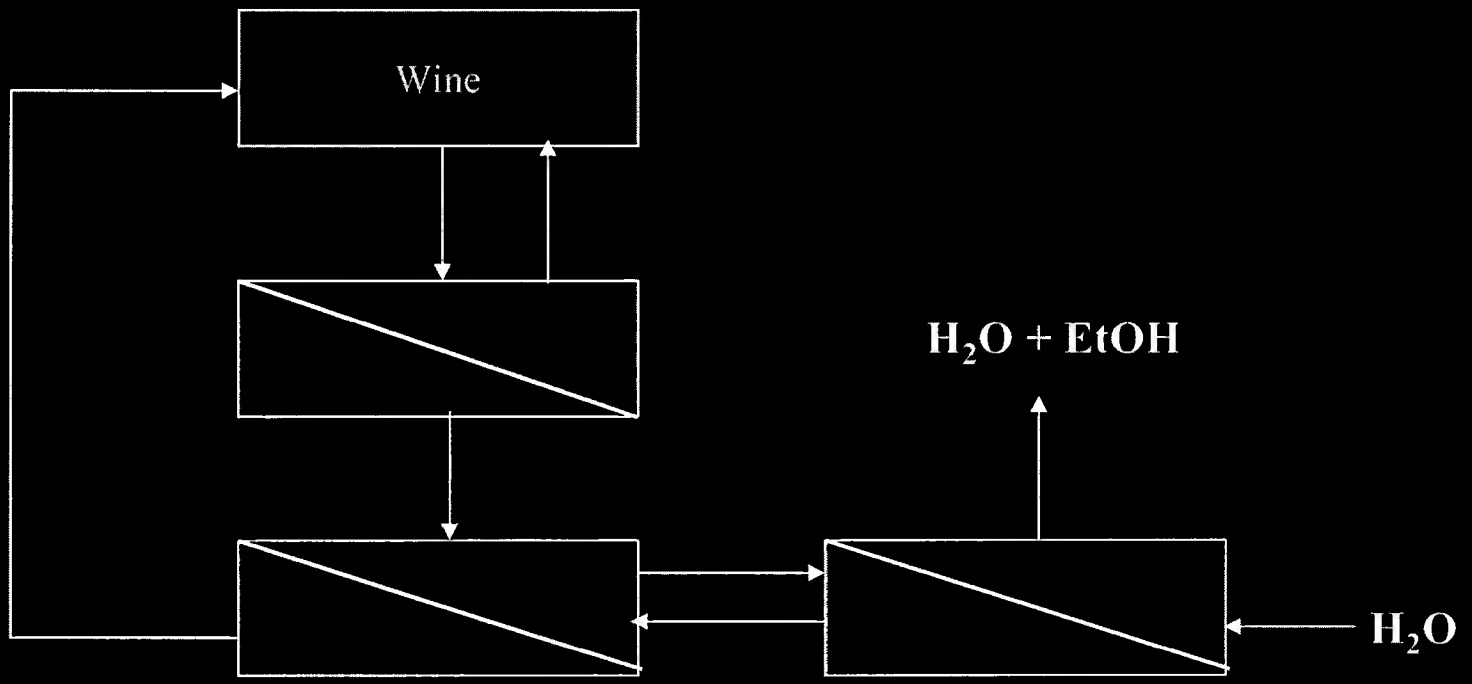
TO AVOID LOSS OF FLAVOR





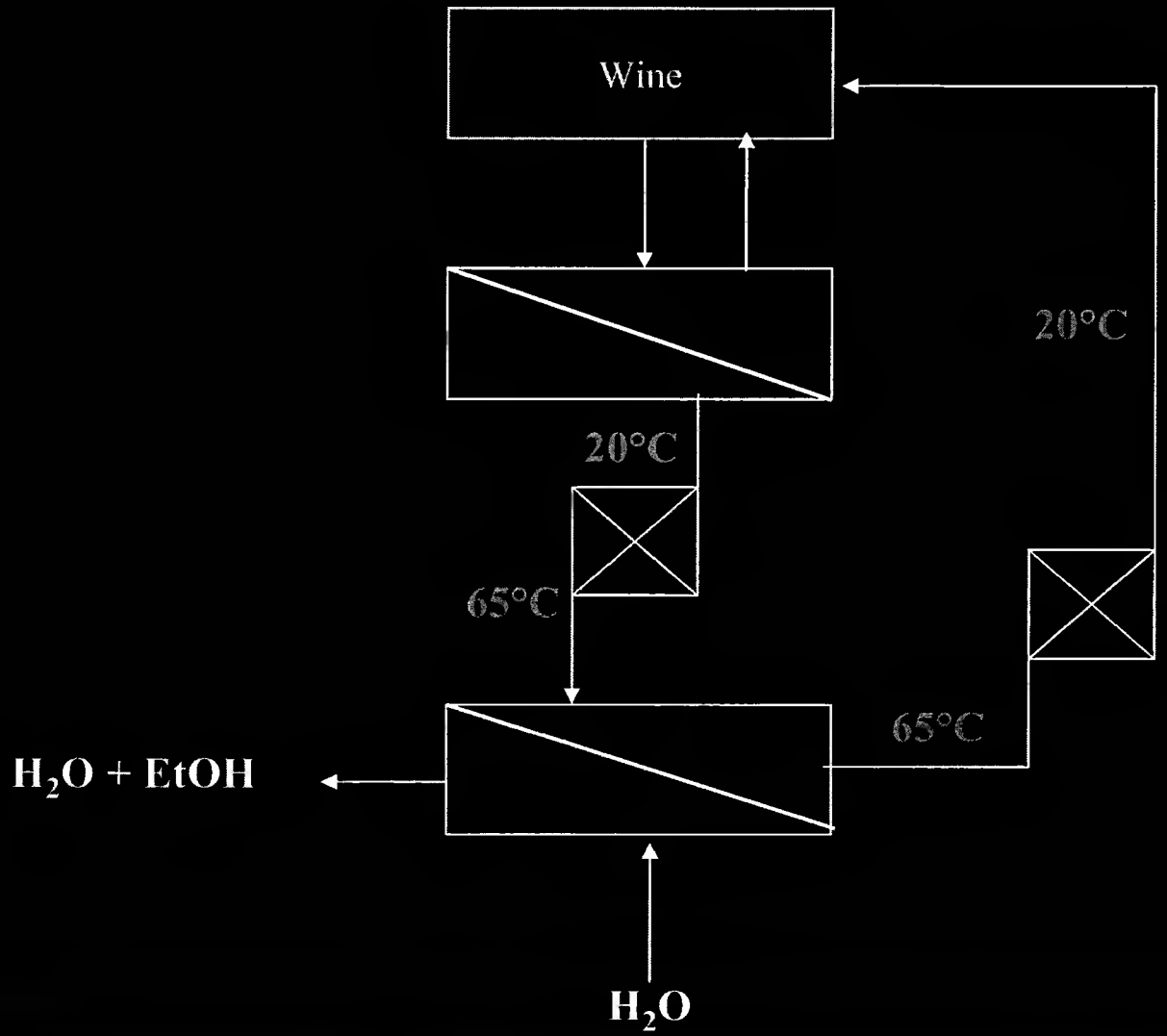
2

TO REDUCE THE CONSUME OF WATER



3

TO IMPROVE YIELDS



Results and considerations about "membrane contactor" techniques

- This technology allows a partial dealcoholization with quality results similar to other techniques.
- Compared to other techniques currently evaluated by OIV, it permits partial wine dealcoholization with only one treatment and without splitting the wine.
- This technique, in conjuction with anionic resins, can also be used to remove some other volatile compounds from the wine, for example acetic acid: however the treatment doesn't require wine splitting because the acetic acid elimination occurs in the external environment of the product (extractant solution).

Results

- To communicate the dealcoholization on the label gives an higher value to the wine for the costumer
- There are contraddictory effects concerning the sensory profile (mainly with strong dealcoholizations …)
- Other studies of the same authors highlights the complexity of the dealcoholization effect and its relation with the type of wine, the tratment rate...

When to deacoholize?

- To re equilibrate a wine
- To produce a low alcohol content wine, always respecting the minimal alcohol content required by law: it's necessary to understand which is the lowest alcohol content that still characterize a wine under the sensory and cultural profiles
- To produce new beverages from grapes

Conclusions

- Dealcoholization, more than an opportunity, is often a necessity also in the production of a “tradictional” wine
- Certainly dealcoholization can be an opportunity considering the present purchases junction and the social needings concerning the alcohol intake
- The technique is interesting on te “Prosecco”…

Acknowlegments

- Vason Group / JU.CLA.S. - Italy
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- Collaboratori DiSTeMeV: C. Carbognin, E. Nicolis, E.M. Casarotti, M. Manzo, N. Righetti

INTERNATIONALER IVIF-KONGRESS 2010

24.–28. MÄRZ 2010 NEUE MESSE STUTTGART

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Alkoholmanagement

Alcohol Management

24.03.2010

International Congresscenter Stuttgart

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Internationale Technologiemesse für Wein, Obst, Fruchtsaft und Spirituosen

INTERVITIS INTERFRUCTA

Neue Messe Stuttgart 24.–28. März 2010

Alcohol management in the winery

David Wollan, Memstar Pty Ltd dwollan@memstar.com.au **(Copyright)**

IVIF 2010 - Session Wednesday 24th March 2010 (WAB1)

Introduction

Other speakers will, no doubt discuss the clear trend to increasing alcohol concentrations in wines all over the world. They will speculate on the origins of this phenomenon and its effect on the sensory properties of different alcohol levels in wines. I am sure they will also suggest viticultural and microbiological solutions to the problem. My brief is to discuss some more technological (specifically physico-chemical) approaches to the management of wine alcohol. In another presentation, later in the conference I will concentrate more on some specific techniques. For now this will be a brief survey of things the winemaker can do to make wines lower in alcohol.

Options for lower wine alcohol

The options for the winemaker are relatively obvious:

1. Pick grapes earlier. Obviously if the winemaker can achieve satisfactory flavour and tannin ripeness before sugar levels are excessive then they should harvest earlier. This means grapegrowing practices must be excellent and there must also be sufficient resources in the production chain to allow all the grapes to be harvested optimally. In the current climate this is not so easy.
2. Wait for new yeast strains which produce less alcohol. There is tremendous work being done around the world on this but without genetic modification of the yeast there are limits to what can be achieved.
3. Manipulate the composition of must or wine. In the short to medium term, the best prospect for the winemaker is to use some technological process to manipulate the composition of the must or the wine made from it.

Reduce must sugar

The most obvious approach is to reduce the sugar concentration in the must. After all alcohol levels have been manipulated (upward) by the process of chaptalisation for many years. If sugar concentrations could be reduced then this would, of course, lower wine alcohol. However this is not so easy to achieve in practice.

Add water / Dilution

Adding water to must is a common practice around the world. It:

- reduces sugar concentration, but
- also dilutes other, desirable wine components such as acid, flavour, tannin, colour etc.

- is illegal in most countries around the winemaking world. Water addition is generally regarded as cheating with a poor effect on wine quality. Interestingly, this is not the case in the USA where some dilution to 22°Brix is permitted.

Bucher "Redux" process

There is a proprietary, patented process for reducing the sugar concentration of must. It is offered by the Bucher company. It is called "Redux" and will be discussed later in this presentation.

Options to reduce wine alcohol

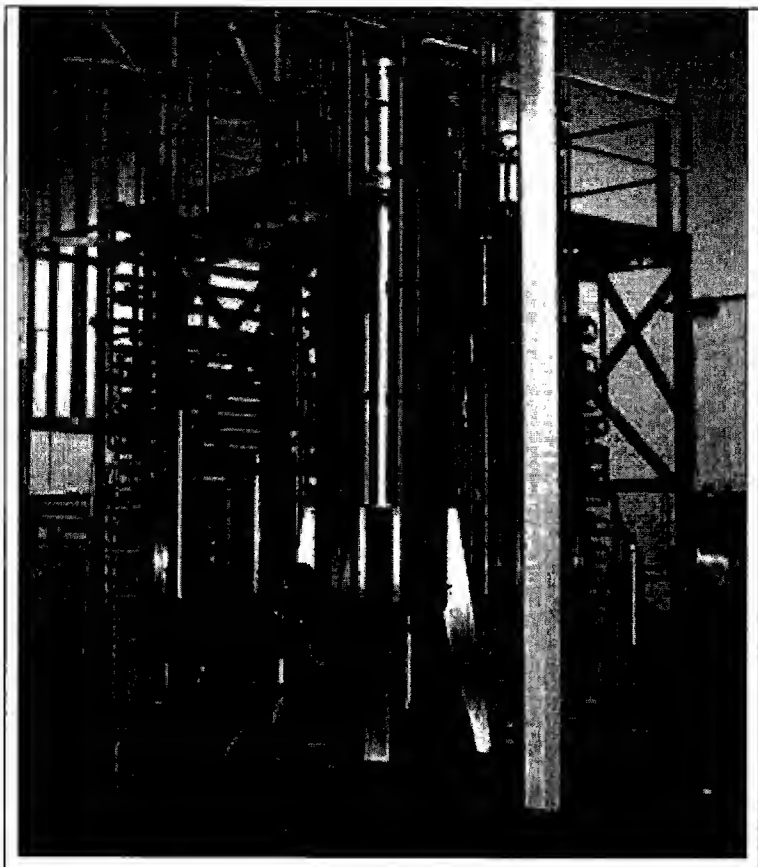
If the must composition is not adjusted before fermentation then the alcohol composition must be adjusted later. As with grape must, it is possible to add water and so dilute the wine but this raises many of the same objections as the dilution of must so it will not be considered further. In fact I am not aware of any major wine producing country which permits this practice.

If the wine alcohol concentration is not to be reduced by dilution then the alcohol must be removed from the wine, while hopefully removing few other desirable components. Currently this is achieved using two broad categories of processes:

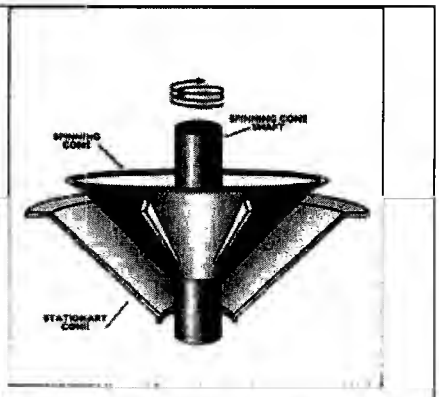
- Distillation based techniques rely on the different boiling points of alcohol and other wine components then uses fractional distillation techniques to separate them.
- Membrane based techniques rely on the different permeabilities of alcohol and other wine components for a given membrane or membranes.

Spinning cone distillation

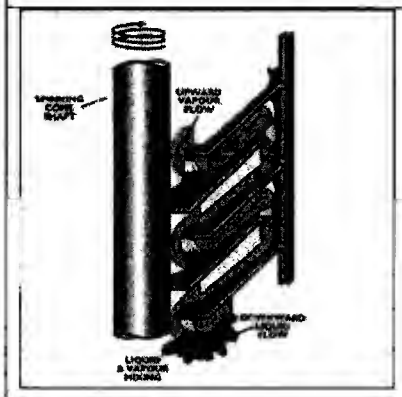
Of the various distillation techniques, the most successful and widespread is the Spinning Cone Column. Essentially the process is based on a column with a stack of alternating static and spinning cones. Product enters at the top and a thin film forms on the cones as it descends. In counter-flow with this is a stripping stream of vapour (gas or steam) which ascends the column under vacuum. Volatiles such as flavour and alcohol are thus removed. In the case of alcohol adjustment, a portion of the wine is processed through the column twice. On the first pass at lower temperature (~25°C) delicate flavours are removed and stored. On the second pass at higher temperature (~40-50°C), alcohol is removed and the flavours and de-alcoholised wine are then recombined with the bulk of the wine.



Spinning Cone Model SCC10,000 (FT Technologies, 2009)



Spinning cone - cut away



Spinning cone – liquid-vapour flow path

This system is complex, capital intensive and relatively immobile. There is also some possibility of flavour loss or damage, and the alcohol is removed at relatively low strength (approximately 55-60% v/v) so overall volume loss from the wine is significant. On the other hand, it is capable of reducing the alcohol in wine to a very low level in just two passes so it is relatively quick and efficient.

Membrane options

There are a number of membrane based separation technologies which are relevant to the reduction of wine alcohol:

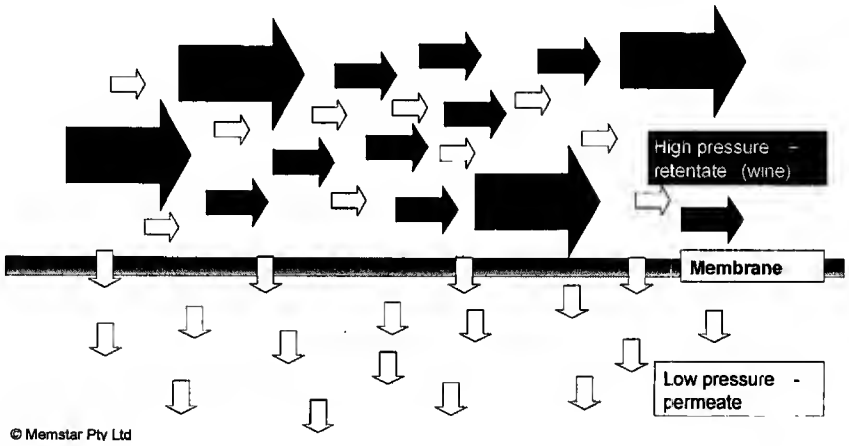
- *"Reverse osmosis: liquid-phase pressure-driven separation process in which applied transmembrane pressure causes selective movement of solvent against its osmotic pressure difference."* (International Union of Pure and Applied Chemistry, 1996)

- *"Nanofiltration: pressure-driven membrane-based separation process in which particles and dissolved molecules smaller than about 2 nm are rejected." (International Union of Pure and Applied Chemistry, 1996)*

- (Evaporative) *"Perstraction: separation process in which membrane permeation and extraction phenomena occur by contacting the downstream with an extracting solvent" (International Union of Pure and Applied Chemistry, 1996).* This process is sometimes referred to as "osmotic distillation" but this is not really correct as osmosis is not the driving forces and distillation, in the traditional sense, does not occur.

- *"Pervaporation: Membrane-based process in which the feed and retentate streams are both liquid phases while permeant emerges at the downstream face of the membrane as a vapour." (International Union of Pure and Applied Chemistry, 1996)* I mention this here because it has some application in bio-ethanol production but its application to winemaking is theoretical only at this stage.

Membrane separation - Reverse osmosis and nanofiltration



Reverse osmosis and nanofiltration have in common the process of pumping wine at high pressure in a cross flow type configuration past a semi-permeable membrane. The membrane - reverse osmosis (RO) or nanofiltration (NF) - allows small molecules in the wine to permeate or pass through it differentially according to their size.

The wine remains substantially on the high pressure or 'retentate' side of the membrane and its vinous character is retained in the unfiltered portion which becomes concentrated. Some of the lower molecular weight fraction passes slowly through the filter membrane to the low pressure, 'permeate' side. This permeate has the appearance of water and contains little if any of the colour or flavour of wine.

Some wine components' molecular weights

Water	18	
Carbon dioxide	44	
Acetaldehyde	44	
Ethanol	46	
Acetic acid	60	
Ethyl acetate	88	
Lactic acid	90	"tight" RO↑
Glucose / Fructose	180	
Flavonoids	>300	

This table of various wine components and their molecular weights shows that this ability to fractionate wine according to molecular weights in the range of 50 to 300 is a very powerful tool. For significant passage, typical RO membranes have a nominal molecular weight (MW) cut-off (MWCO) of 100 Daltons. They allow the passage of only those components to be removed: water (MW=18) is the smallest molecule in wine and passes readily. Other constituents with MW<100 such as carbon dioxide (MW=44), acetaldehyde (MW=44), ethanol (MW=46), acetic acid (MW=60) and ethyl acetate (MW=88) are passed into the permeate but at lower concentrations than are present in the wine. Almost all other constituents including the main wine acids, sugars, esters, anthocyanins and other phenolics do not pass the membrane at all and are retained in the wine.

Nanofiltration membranes are differentiated from RO membranes by their permeability. The type of nanofiltration of interest in the wine industry is really a particular type of 'loose' reverse osmosis where the molecular weight cut-off of the membranes is between about 150 and 300 Dalton. Practically, these membranes are used in preference to "tighter" reverse osmosis membranes when higher permeate flux rates and/or greater passage of higher molecular weight components are important. In all such choices there is inevitably some compromise between the passage into the permeate (and potential loss) of desirable wine components as well as undesirable.

Reverse osmosis does not reduce alcohol!

There is a common misunderstanding that reverse osmosis or nanofiltration can reduce wine alcohol. This is sometimes encouraged by ignorant or unscrupulous vendors. In my long experience working in this area, it has always been the case that:

By itself, RO increases the concentration of alcohol in treated wine.

Some secondary treatment is essential to remove the alcohol from the permeate stream.

Comparison of concentration effects - Different membrane types

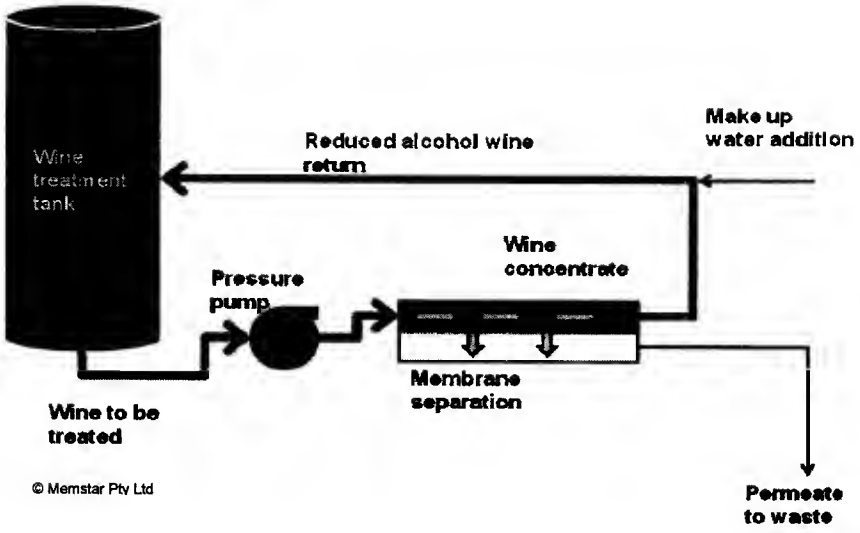
The following table illustrates the effect of concentrating wine with two different membrane types. In both cases the concentrated wine increased in alcohol concentration.

	Titratable Acidity (g/l as tartaric acid)	Alcohol (% v/v)
Control wine	5.9	14.10%
8% Concentration with "loose", NF type membranes:-		
Permeate	2.4	13.35%
Concentrate	6.2	14.15%
8% Concentration with "tight", RO type membranes:-		
Permeate	0.9	8.00%
Concentrate	6.3	14.70%

Reverse osmosis and water addition

One option to overcome this fact is to use RO (or NF) to remove a volume of alcoholic permeate and replace it with a similar volume of water. Besides the fact that in most places this is illegal, while permeate is not particularly vinous, it does contain a number of desirable components. This will depend on the type of membrane used but, in general these desirable wine components will be lost if the permeate is removed and replaced with water.

It is not a strategy to be encouraged.



Membrane separation, treatment and recombination

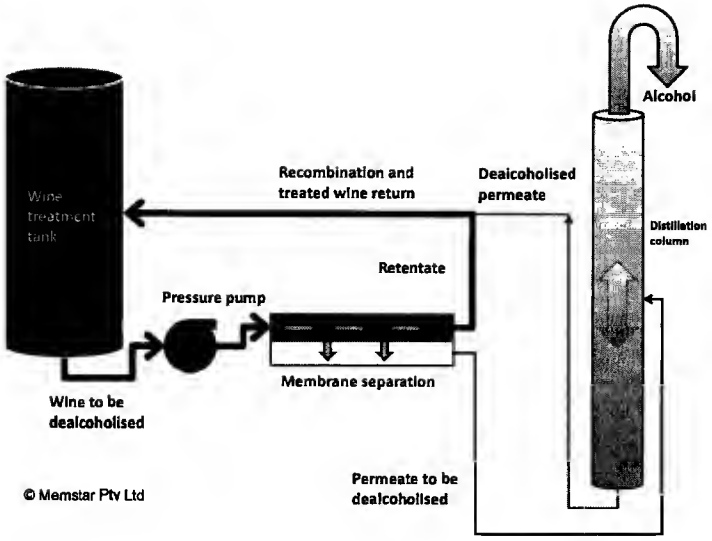
There are a number of legitimate membrane techniques that are being used for reducing wine alcohol and all involve at least two stages. Broadly, the strategy can be described as membrane separation of wine into a permeate and concentrate scheme; the treatment of the permeate to remove some or all its alcohol and the recombination of the dealcoholised permeate with the wine concentrate.

The reason for this strategy is that most of the sensitive and desirable wine components stay in the concentrate and so are not damaged or lost in the second, alcohol removal stage.

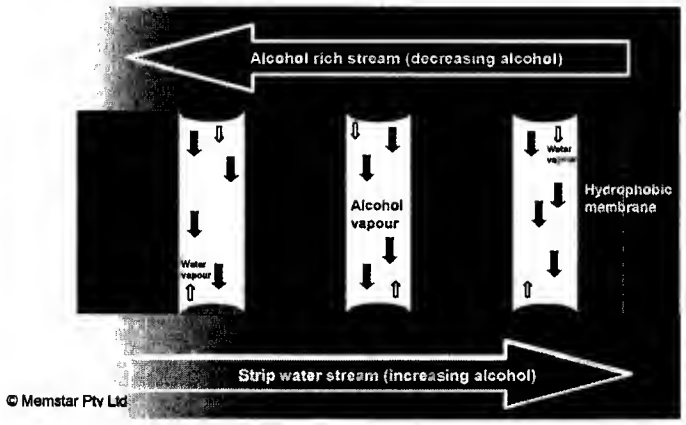
Membrane separation and permeate distillation

The first example of such a two stage technique involves membrane separation of a permeate stream which is distilled in a fractionation column. The alcohol is removed from the permeate and the dealcoholised portion is returned to the wine.

The main point to note is that if the membrane separation is with NF membranes there are a number of heat labile components (e.g. sugars) in the permeate that could be damaged in the distillation process. In this case, it is preferable to use vacuum distillation to keep the temperatures down. If RO membranes are used, the permeate is less heat sensitive and conventional distillation may be used.

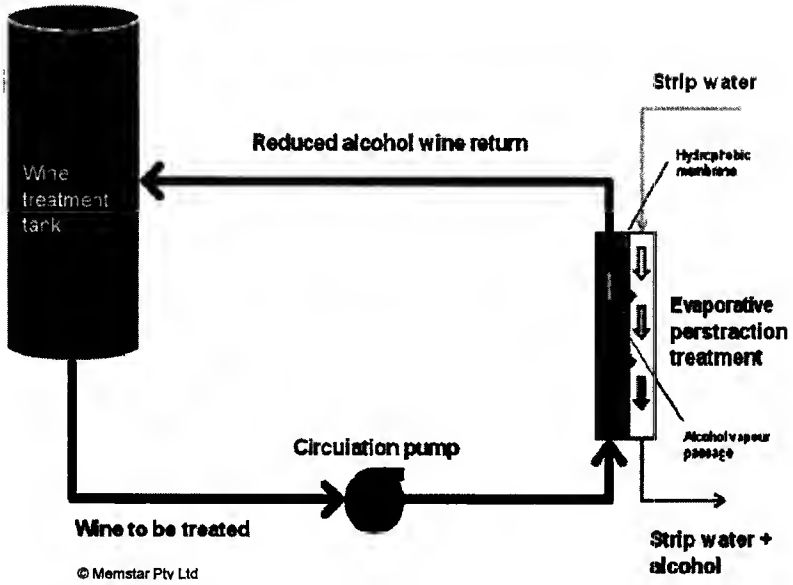


Evaporative perstraction



There is another single step membrane separation technique that should be explained at this stage. This technique involves passing a stream of wine through a membrane contactor. Separated from the wine stream by a hydrophobic membrane, a counter-flow of water is passed through the same contactor and alcohol passes through the membrane from the beverage to the water. This process is based on the principle that ethanol, as a volatile wine component, has a significant vapour pressure. This leads to its movement into the porous matrix of the hydrophobic membrane and by virtue of the concentration difference across the membrane, its subsequent condensation and dissolution into the water on the other side.

Direct evaporative perstraction (Osmotic distillation)



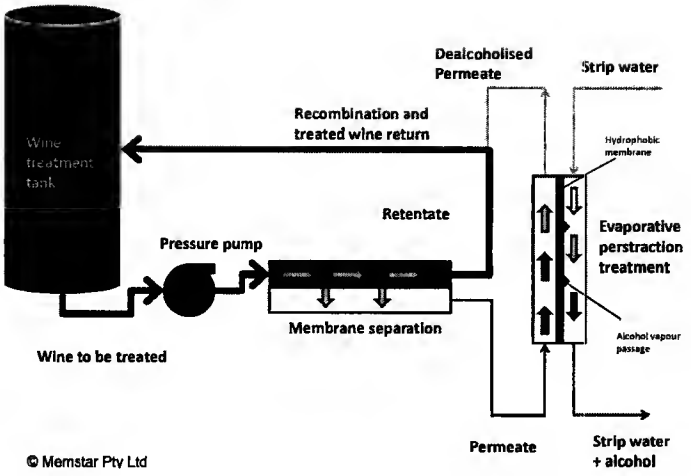
In practice, direct perstraction treatment of wine leads to high levels of extraction of other desirable volatile components from the wine, such as flavours, esters and sulphur dioxide.

Another characteristic of this particular process is the undesirable tendency for water vapour to pass across the membrane from the strip water to the wine. This occurs because the vapour pressure of water above the strip water surface is slightly higher than the water vapour pressure on the wine side of the membrane as a result of the higher osmotic potential of the wine. This trans-membrane migration limits the practical application of direct treatment of wine using evaporative perstraction.

Despite some of the problems associated with this technique, it is offered commercially and consists of an arrangement more or less as above. Ideally, (but not shown here) the system should allow for pre-treatment of the strip water to deoxygenate, dechlorinate and remove possible taints that might migrate into the wine.

Membrane separation and evaporative perstraction

My company Memstar has overcome the inherent problems of direct perstraction (or osmotic distillation) by applying the process of evaporative perstraction to the permeate stream of reverse osmosis or nanofiltration processed wine. As with previously described membrane separation, treatment and recombination techniques, RO or NF separation of wine into concentrate and permeate streams is followed by a secondary treatment of the permeate. Water is used to strip alcohol from the permeate stream of reverse osmosis processed wine. This permeate is then recombined with the wine from which it was extracted, thus lowering the alcohol of the blend.



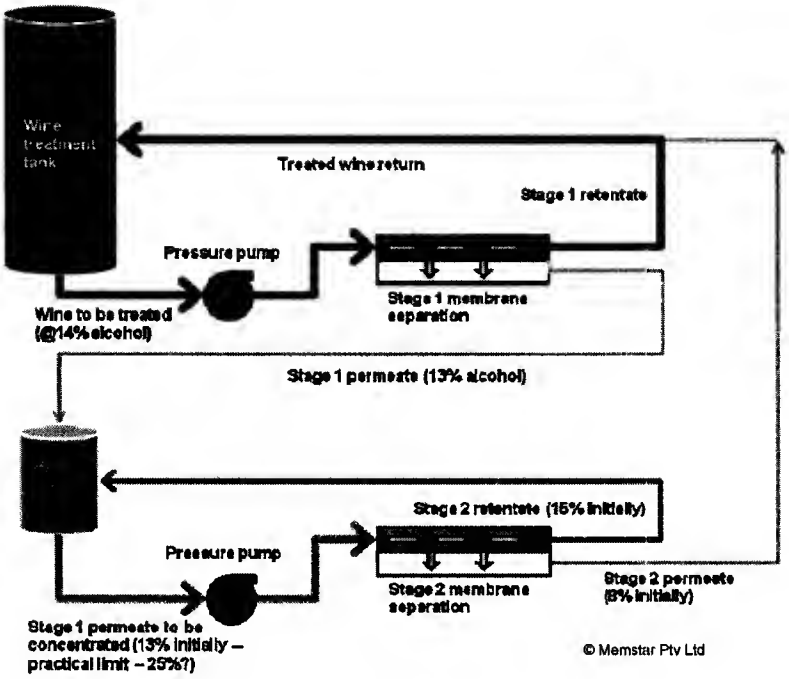
By treating membrane permeate and not wine, the loss of volatiles is substantially reduced because of their limited passage from the wine into the permeate stream. As well as this, the permeate has a much lower osmotic potential than the original wine so minimising the water vapour pressure difference between alcoholic permeate on one side of the membrane and strip water on the other. The result is that any tendency for water to move from the strip into the permeate (and hence the wine) is greatly reduced or eliminated.

This process is controlled and optimised by the selection of appropriate membranes and operating parameters such as temperature, pressure and flow rate to maximise the passage of ethanol while limiting the passage of other compounds. This technology is now well established and has been adopted commercially in many countries around the world. The equipment is mobile and efficient and allows winemakers very fine control of alcohol levels without the complications and expense of more elaborate distillation procedures.

Two stage membrane separation - the "molecular sieve".

Another strategy we experimented with some years ago involves a two stage membrane separation. In the first stage a nanofiltration type membrane is used to create a permeate stream which is then concentrated by a second, reverse osmosis type membrane. The idea is that the first membrane readily passes alcohol and the second membrane has very limited alcohol passage.

We call this approach the "molecular sieve".



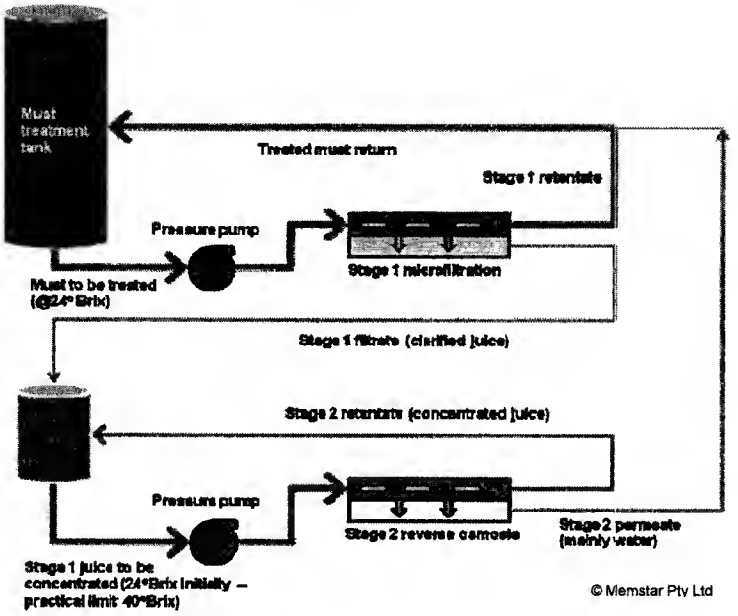
Problems with 2 stage separation technique

Our experience with this concept is that it is:

- Slow, expensive and inefficient
- Large losses
- Membranes with good alcohol separation characteristics are not readily available
- Osmotic pressure of a 25% alcohol solution >100 atmosphere!

We abandoned this approach because it was not successful. Nevertheless we have seen promotional material from a vendor offering such a process. If they have overcome some of these problems, we will stand corrected.

Sugar reduction of must (Bucher Redux®)



The "molecular sieve" principle of 2 stage separation has been applied to reducing must sugar concentration in the proprietary Redux® process offered by Bucher. In this case, the first stage involves a cross flow microfilter and the second stage is a reverse osmosis separation. We have not been able to find much published detail of this process but from available information and our own experience, we believe it will suffer from some of the same limitations as the molecular sieve approach to alcohol reduction. In particular, the practical concentration limit for the reverse osmosis step is about 40°Brix so the eventual by-product in the buffer tank will be a low strength grape juice concentrate. Unless the winemaker has other uses for this material such as enriching low sugar musts or sweetening finished wines then it will represent a relatively large volume loss.

Getting the alcohol right

We have surveyed a number of different technologies which can all remove alcohol. These have different tendencies to remove or damage other components (and add undesirable components). But is there a "Right" alcohol concentration? And how easy is it to achieve, practically?

Finding the "Sweet Spot"

Over many years of practising alcohol adjustment, we are convinced that there are certain points where the different alcohol concentration results in a wine where the expression of wine flavour and balance is most satisfying. We call these points "sweet spots" and recommend to all winemakers practising alcohol reduction that they understand when and how to find and achieve these. In our experience, alcohol reduction to a "recipe" or formula can be disappointing because the difference between a sweet spot and not may be as little as 0.1% alcohol.

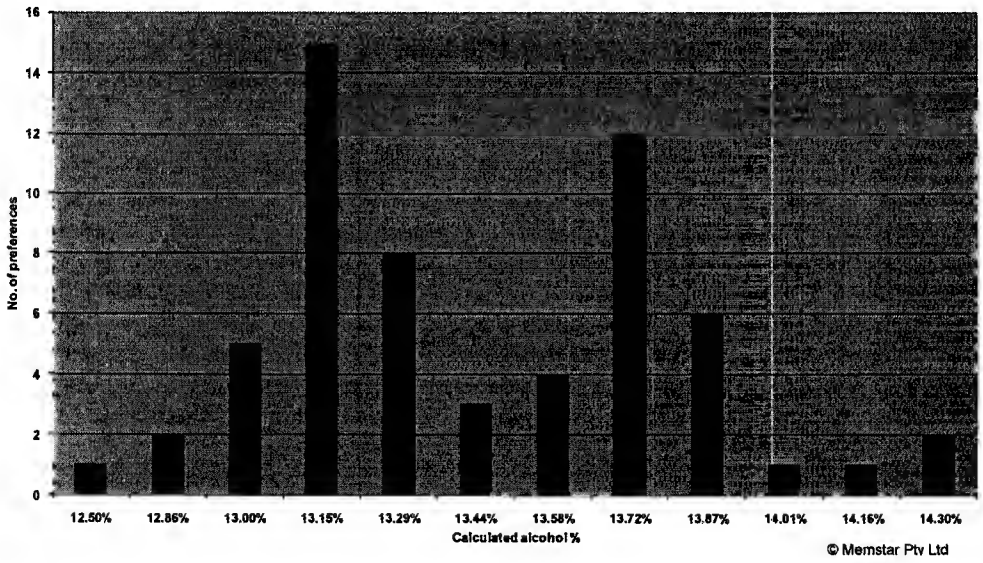
Sweet spot methodology

We have developed a procedure for doing this but other winemakers will have their own approaches:

- Blend high and low alcohol samples incrementally
- Bracket a likely range of alcohol concentrations with incremental changes of about 0.1% to 0.2%
- Select one or two wines that are preferred

Sweet spot histogram

If we have a large tasting group we can collate, chart and display the tasting results on a frequency histogram. When we have done this it is not uncommon to have a result where there is more than one favoured wine. These are often distinctly different styles and it becomes a matter for winemakers to decide which best suits their requirements.



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Neue Messe Stuttgart 24.–28. März 2010

Technological options for dealing with high alcohol wines

Technische Optionen bei der Behandlung von alkoholstarken Weinen

David Wollan, Memstar Pty Ltd dwollan@memstar.com.au **(Copyright)**

IVIF 2010 – Session Saturday 27th March 2010 (WB5)

Abstract

Over recent years around the winemaking world there has been a steady and significant increase in wine alcohol concentration. Some of this observed rise can be attributed to changes in viticultural and winemaking fashion and practices but undoubtedly climate change is a major and continuing contributor.
For winemakers this has been a worrying trend which poses numerous challenges. They need to find methods to limit or reduce the alcohol content of their wines while meeting the expectations of consumers and regulators. While some modifications in practice may have a marginal effect, only technological intervention has any real prospect of solving this problem. Different jurisdictions take different approaches to the regulation of these processes but the urgency of the problem means that winemakers have already started considering the alternatives.
This paper will survey a number of options for reducing alcohol. These include approaches such as basic water addition, the use of low alcohol forming yeast, membrane based systems such as reverse osmosis and nanofiltration (alone or in combination with other techniques such as distillation), and distillation based systems such as the spinning cone column.
These techniques will be assessed for their impact on wine quality, integrity and cost.

Kurzfassung

Weinproduzenten sehen sich in den letzten Jahren weltweit mit einem kontinuierlich und deutlich ansteigenden Alkoholgehalt im Wein konfrontiert. In einzelnen Fällen mag der erhöhte Alkoholgehalt auf veränderte önologische Produktionsmethoden zurückzuführen sein, zweifellos trägt jedoch der Klimawandel zu einem hohen und kontinuierlichem Maß dazu bei.
Winzer beobachten diese Entwicklung mit zunehmender Sorge und sehen sich einer ganzen Reihe neuer Herausforderungen gegenüber. Ziel ist es, Methoden zu entwickeln den Alkoholgehalt im Wein zu reduzieren und dabei gleichzeitig den Erwartungen des Konsumenten und den Gesetzen zu genügen. Da sich die Veränderung einiger praktischer Details nur marginal auf eine Senkung des Alkoholgehalts auswirkt, müssen erfolgsversprechende Lösungen einzig in technologischen Entwicklungen gesucht werden. Die Gesetzgebung verhält sich der Regulierung dieser Prozesse gegenüber unterschiedlich, der Dringlichkeit des Problems Rechnung tragend, haben jedoch einige Winzer damit begonnen, über Alternativen nachzudenken.

Dieser Vortrag soll einen Überblick über einige Möglichkeiten zur Alkoholreduktion geben. Dazu gehören Verfahren wie Wasseraddition, die Verwendung von Hefen mit geringerer Alkoholbildung, Membranverfahren, wie die Umkehrosmose und Nanofiltration (alleine oder in Kombination mit anderen Techniken, wie zum Beispiel der Destillation), sowie Systeme auf Destillations-Basis wie die Spinning Cone Column (Schleuderkegeltechnologie).
Die Auswirkungen dieser Technologien in Hinblick auf die Weinqualität, Fehlerfreiheit und Kosten sollen beurteilt werden.

Introduction

In my earlier presentation to this conference I surveyed the problem of high alcohol wines and looked at the various techniques for managing this in the winery. In this presentation I will look more closely at some practicalities around specific technological options. In particular I will examine the technology with which I am personally most familiar.

To restate the obvious, winemakers want:

- the benefit of fully ripe grapes without high alcohol
- to remove alcohol only
- to aim for the ideal alcohol level

Assuming that this will involve a technological intervention, the process should:

- be acceptable to legal and regulatory authorities
- be economically viable
- have minimal environmental impact
- be acceptable to consumers

Separating alcohol from wine

So the challenge for any process is to be able to separate alcohol from wine without removing or damaging other wine components. Traditionally alcohol has been extracted from wine by fractional distillation. This is based on the different boiling points of ethanol, water and other wine components. At atmospheric pressure, various wine fractions boil over a range of about 78°C to 100°C. At these temperatures, many wine components are damaged, creating off-flavours. Also, many desirable components have boiling points very similar to alcohol so are difficult to separate from the alcohol which is removed. When the primary purpose of distillation was to extract the alcohol as a valuable product – spirit or brandy – any harm done to the wine was irrelevant.

Distillation – Spinning cone column

The task being considered here is to preserve the inherent character of the wine while taking out the alcohol. Lower temperature distillation with better fractionation is critical. In this regard, the spinning cone column has been the most successful of the distillation techniques. It operates under vacuum at much lower temperature and preserves wine flavour.

In practice, it is difficult to control the degree of dealcoholisation as wine passes through the column so winemakers treat a small portion of the wine (10% - 30% depending on the extent of treatment desired). This portion is virtually completely dealcoholised and then blended back with the bulk of the wine.

Because of the capital cost and relatively immobile nature of the equipment, it is unusual for wineries to own their own plant. Unless wineries are very large and can justify the cost, they typically send a portion of their wine to be dealcoholised at fixed plants. While the actual cost of the process is not high, logistical complexities and other costs can make it a less attractive option.

Alcohol removal by membrane separation

For many winemakers, a more accessible approach is to use membranes to separate alcohol. This is based on differential permeabilities of alcohol and other wine components for a given membrane type. And in this regard, there is no single membrane technique which will selectively pass alcohol and alcohol only. Therefore a combination of techniques is required.

Generally the processes people think of when discussing membrane separation are reverse osmosis (RO) and nanofiltration (NF). In my earlier presentation I explained these in broad detail but I will repeat a few key points:

- RO and NF do not reduce alcohol (a common misunderstanding)
- By itself, RO (and NF) increases the concentration of alcohol in treated wine
- Some secondary treatment is essential

The following table comparing concentration effects for different membrane types illustrates this:

	Titratable Acidity (g/l as tartaric acid)	Alcohol (% v/v)
Control wine	**5.9**	**14.10%**
8% Concentration with "loose", NF type membranes:-		
Permeate	**2.4**	**13.35%**
Concentrate	**6.2**	**14.15%**
8% Concentration with "tight", RO type membranes:-		
Permeate	**0.9**	**8.00%**
Concentrate	**6.3**	**14.70%**

In my previous presentation I explained the following membrane options which I will not discuss further on this occasion:

- RO (/NF) and water addition
- RO (/NF) and permeate distillation

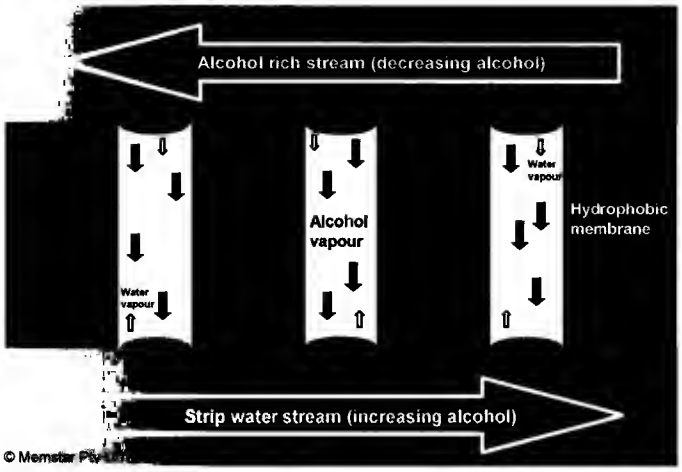
I also explained the following processes:

- Evaporative perstraction (Osmotic distillation)
- RO (/NF) and evaporative perstraction

But I will now look at these in more detail.

Evaporative perstraction

Briefly, this process involves the migration of alcohol across a hydrophobic membrane, driven by vapour pressure difference across the membranes as a result of different alcohol concentrations on each side of the membrane.



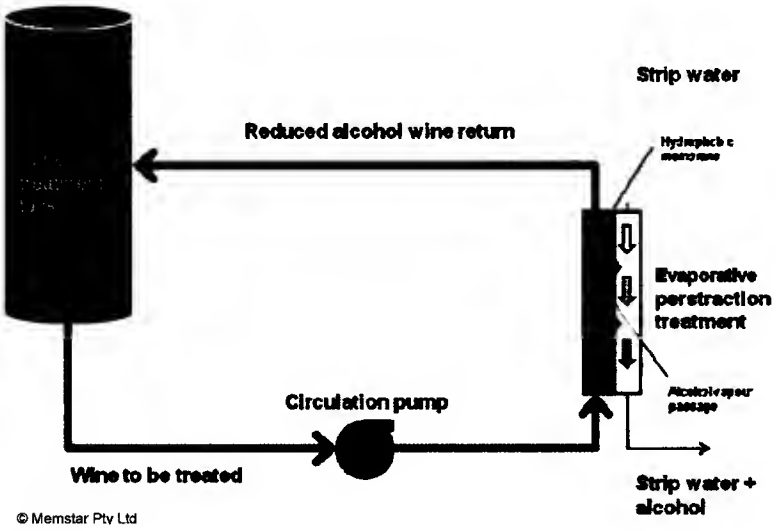
In practice, the rate of alcohol removal varies with:

- Alcohol concentration difference between sides of the membrane
- Membrane surface area
- Temperature

Direct evaporative perstraction (Osmotic distillation)

In the USA, this technique came to be called Osmotic Distillation even though the original inventors referred to it as Evaporative Perstraction.

The first described use goes back to 1993 and involved the circulation of wine through a hydrophobic membrane contactor.



The process was recognised to have some deficiencies by the inventors. Of most relevance are:

- Loss of volatiles
- Water transfer into wine
- Membrane fouling

In particular, when 9% alcohol wine was directly contacted with a strip solution of varying composition, the relative flux of water and alcohol could be manipulated according to the alcohol and salt content of the strip water. In trial number 1 below, for plain strip water there was a negative water flux i.e. water flowed into the wine. This could be overcome by increasing the osmotic pressure of the strip water relative to the wine by adding 7% NaCl (trial number 2).

Flux across a hydrophobic membrane of alcohol and water from a 9% alcohol wine

Number	Strip Alcohol %	Strip NaCl %	Alcohol flux ($kg/m^2.hour$)	Water flux ($kg/m^2.hour$)
1	0	0	1.0	-0.2
2	0	7	1.0	0
3	0	27	1.0	1.38
4	3	0	0.54	-0.2
5	3	7	0.4	0
6	3	27	0	1.38

(Michaels, A.S., Membrane Extraction Process; Patent application, International Publication Number WO 93/22036 WIPO 1993)

Membrane separation, treatment and recombination

My company, Memstar, overcame these deficiencies by applying the process of evaporative perstraction to the permeate separated in a previous membrane

treatment. This is another example of the membrane separation, treatment and recombination principle I have described previously.

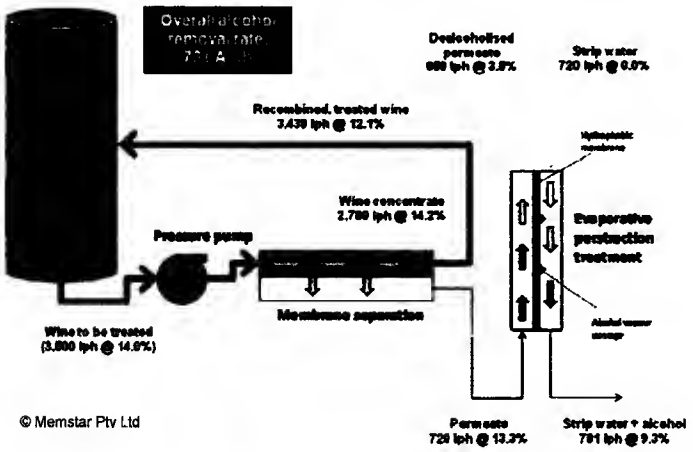
Membrane separation and evaporative perstraction

The following illustration shows the key details of this process. It refers to the typical operating parameters of a particular, mid range model ("Midi 8") that currently operates commercially.

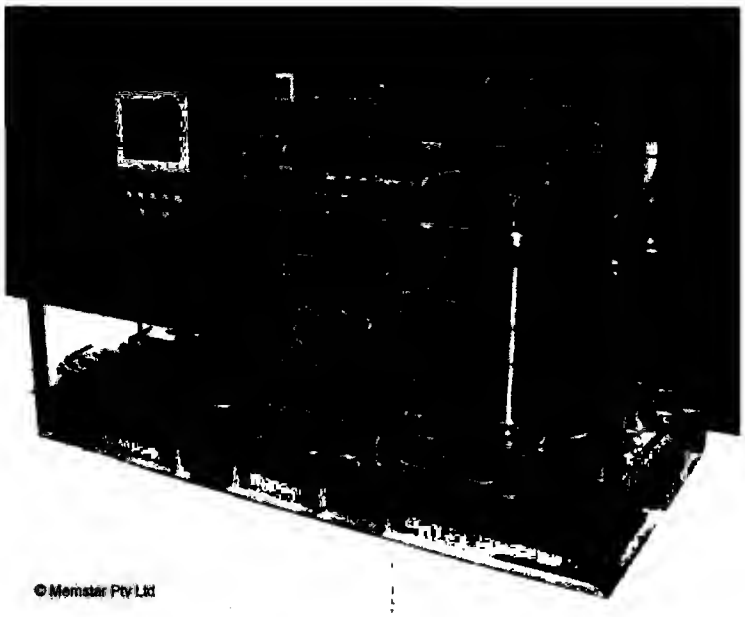
Wine at alcohol 14% is pumped through a membrane separation stage at the rate of about 3,500 litres per hour (lph). A permeate stream of about 13.3% alcohol passes through the membranes at the rate of about 720 lph. This leaves a 14.2% concentrate stream to recirculate back to the treatment vessel at the rate of 2,780 lph.

The alcoholic permeate then passes through the perstraction stage of the process where it is dealcoholised by a 720 lph stream of strip water. This becomes alcoholised and passes to waste or recovery by distillation at a strength of about 9.3% alcohol. The treated permeate is reduced to about 3.5% alcohol and this is recombined with the wine concentrate to give a stream of treated wine with an alcohol concentration of 12.1% (a reduction of 1.9%).

In the configuration shown, the treated wine is returned to the original vessel but it could also pass directly to a treated wine tank if this is sufficient treatment. Anyway, the overall effective removal rate of alcohol for this particular system configuration calculates to be 73 litres of alcohol per hour (LAL/h).



Memstar "Midi 8"



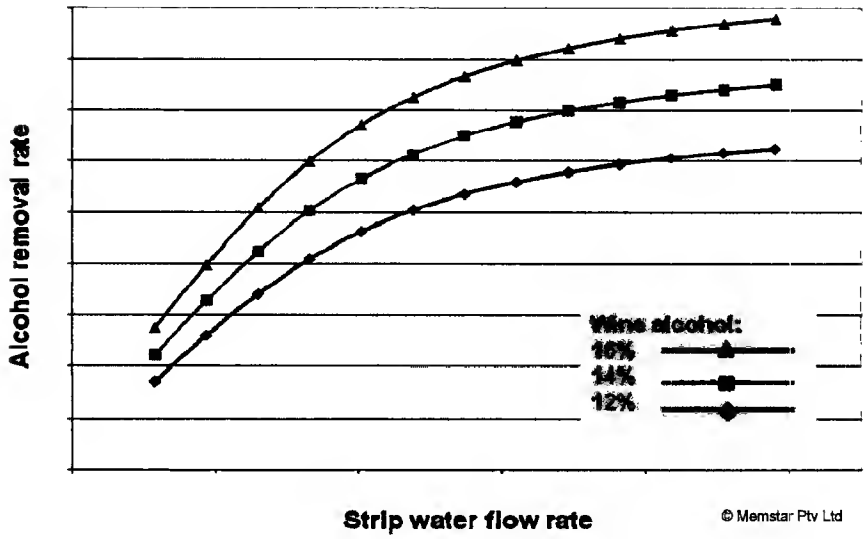
System performance

For a system of given membrane surface area and operating temperature, the main performance drivers are:

- Wine alcohol concentration
- Relative flow of permeate and strip water

Performance parameters – Perstraction systems

The alcohol removal rate under given conditions, will be greater for wines with higher alcohol and increases as the flow of strip water increases. In practice, strip water flow is optimised so that water use is minimised and that the alcohol content of the strip water is maximised.



System design permits these limits to be extended by increasing the membrane area and temperature of processing.

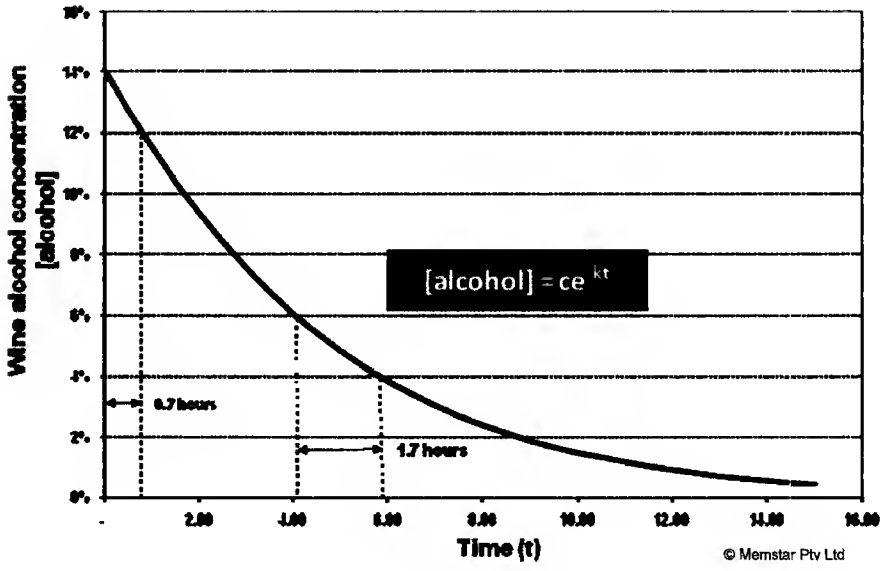
Alcohol reduction over time

Like many membrane systems, it is a characteristic of perstraction systems that the rate of alcohol removal varies with the alcohol concentration.

- **d[alcohol]/dt = c x [alcohol]**

This integrates to give the general function:

- **[alcohol] = ce^{-kt}**



Practically, this means that for a given alcohol reduction, as the alcohol concentration decreases, the time taken increases. For example in the hypothetical situation above, for a reduction from 14% to 12%, the time taken is 0.7 hours. For a reduction from 6% to 4%, the time taken is 1.7 hours.

Consequently there is no advantage in treating a portion of the wine:

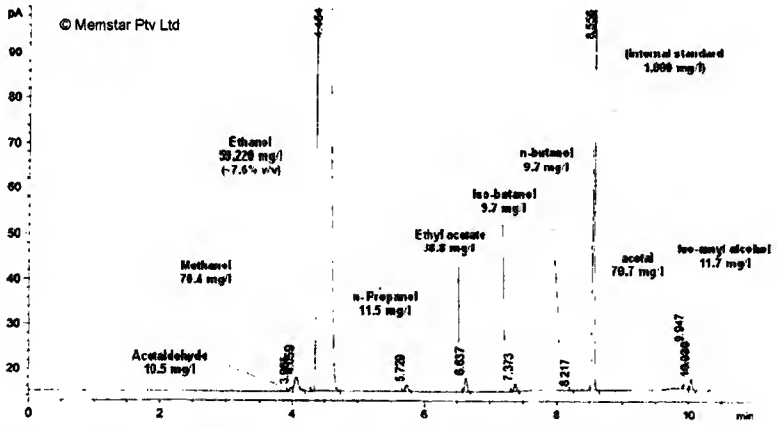
"It is more efficient to take a little alcohol from a lot of wine than a lot of alcohol from a little wine."

In general membrane systems are ideally suited to such trimming operations. It is not practical, or theoretically possible, to achieve a zero alcohol concentration.

Other analytical parameters

Based on many years of experience, we have observed minimal changes in wine components other than alcohol. There is a small drop in sulphur dioxide (~5-10 mg/l) and dissolved CO_2 is reduced. Dissolved O_2 is generally reduced but this also depends on other cellar practices.

GC analysis of typical strip water by-product shows the presence of minor concentrations of other volatile higher alcohols and esters. These components do pass from the wine into the permeate and then into the strip water but at much lower concentrations than in the original wine.



Partial dealcoholisation of wine - regulatory situation

The regulatory situation for the partial dealcoholisation of wine varies around the world. It is now generally accepted as a permitted oenological practice; though there will be specific local regulations governing when, how and which techniques are accepted. The OIV has passed Resolution OENO 10/2004 which permits the practice subject to certain conditions.

In Europe, partial dealcoholisation is permitted and controlled by EC regulation 606/2009 (Annex 1A-40 & Appendix 10). Again there are a number of conditions; the most important of these is that the maximum permitted reduction is 2%.

It appears that none of these regulations is prescriptive about any particular process. (Spinning cone column and reverse osmosis based techniques all seem to be acceptable, with the possible exception of those processes that involve extraneous water addition.)

Economic considerations

Most processes are capital intensive, so are not likely to be feasible purchases for smaller wineries. However in most countries where dealcoholisation is permitted, contractors will provide fixed or mobile dealcoholisation services

The typical cost of this process will vary greatly according to the process, location and scale of operation. Obviously the cost of treating a 20 hl batch will be much greater than a 2,000 hl batch. From experience in a few countries and early reports from European sources, I expect the cost to be in the range 2€ – 10€/hl.

Because different systems can have different proportional wine volume losses, the choice of process can add to this cost.

Environmental considerations

The first key environmental factor is energy use. This will vary considerably according to the process used and scale of operation. For the previous example of the Midi 8 system, electricity use is less than 15kWh/3.5kl (or 0.5kWh/hl). There is also a gas heating facility on this and butane/propane use is less than 3MJ/hl or ~60g/hl of wine treated.

The other factor is the waste or by-product stream. This consists of water, alcohol and some minor components. This is typically collected and recovered for distillation to high grade spirit.

Consumer reaction

Ultimately, the success or otherwise of any winemaking practice depends on consumer reaction. Alcohol reduction is no exception. When I describe my technology to casual acquaintances, the typical response is:

"You take alcohol out of wine? Why would you want to do that?!!"

However there is growing resistance to high wine alcohol levels (particularly among opinion leaders and "gatekeepers"). For at least the past ten years in the USA and Australia there has been a fascination with extremely ripe, high alcohol wine styles. Winemakers who followed this path are now discovering that the trend may well

have passed. Many are trying to pick their grapes earlier but this is often not feasible so they are increasingly looking at the techniques I have described.

While all the processes I have described are "subtractive" (i.e. they don't involve the addition of anything to the wine), among some consumers there is a lingering suspicion of technological intervention. Unfortunately this is a feature of the age we live in. Consumers want the products they purchase to be "natural" and genuine. They are suspicious of additives, manipulations and a general industrialisation of what they consume. On the other hand, they want their wines to be safe, fault free, tasty, consistent, accessible and low priced.

Wine producers around the world have yet to explain satisfactorily to their customers that it is not possible to have it both ways. Winemaking has always used the technologies at its disposal. Who would willingly go back to traditional winemaking without electricity, pumps, refrigeration?

Winemaking has always been the act of intervention in a natural spoilage process. Without intervention there would be no wine, only vinegar.